MARINA DI FRANCESCANTONIO

"EVALUATION OF BOND STRENGTH, POLYMERIZATION STRESS, ADAPTATION IN CAVITY CLASS II AND OTHER PROPERTIES OF RESTORATIVE SYSTEMS"

"AVALIAÇÃO DA RESISTÊNCIA DE UNIÃO, CONTRAÇÃO DE POLIMERIZAÇÃO, ADAPTAÇÃO EM CAVIDADE CLASSE II E DE OUTRAS PROPRIEDADES DE SISTEMAS RESTAURADORES"

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UNIVERSIDADE ESTADUAL DE CAMPINAS

FACULDADE DE ODONTOLOGIA DE PIRACICABA

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Orientador: Prof. Dr. Marcelo Giannini

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Doctorate thesis presented to the Piracicaba School of Dentistry of the University of Campinas to obtain the Ph.D. grade in Dental Clinic in the Operative Dentistry area.

ESTE EXEMPLAR CORRESPONDE À VERSÃO FINAL DA TESE DEFENDIDA PELA ALUNA, E ORIENTADA PELO PROF. DR. MARCELO GIANNINI

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RESUMO

Os objetivos deste estudo foram: 1) caracterizar os elementos químicos e morfologia das partículas de carga de compósitos convencionais e de baixa contração; 2) avaliar a rugosidade superficial (RS) e 3) a sorção de água (SA) e solubilidade (SO) após acabamento/polimento, 24h e após 1 ano de armazenamento de água; 4) tensão de polimerização (TP) dos compósitos; 5) avaliar a adaptação interna (AI) em cavidades Classe II utilizando técnica restauradora incremental e bloco único 24h e um ano de armazenamento em água e 6) a resistência de união (RU) de sistemas restauradores baseados em metacrilatos e silorano após 24 h e após um ano de armazenamento. Quatro resinas compostas foram avaliadas: duas de baixa contração (Filtek Silorane e Aelite LS) e duas convencionais (Heliomolar e Tetric N-Ceram) Para a RU e análise FF, adesivos do mesmo fabricante foram utilizados: Excite para Tetric N-Ceram e Heliomolar; One-Step Plus para Aelite LS e Silorane System Adhesive para Filtek Silorane. Para caracterizar as partículas de carga, uma porção de cada material (n=5) foi imersa em solventes orgânicos e observada por MEV e EDS. Para avaliar a RS, SR e SO (n=10), os sistemas de acabamento e polimento foram usados seguindo as instruções do fabricante. As superfícies polidas foram analisadas utilizando um perfilometro, determinando a RS. Para a SR e SO , as amostras foram armazenadas e a massa de cada amostra foi medida. Os espécimes foram dessecados novamente e a massa da amostra foi determinada. A SR e SO foram calculados a partir dessas medições. Os dados foram analisados por ANOVA e teste de Tukey (p=0,05).TP (n=5) foi determinada através da inserção dos compósitos entre as hastes de polimetilmetacrilato. Os dados analisados por ANOVA e teste de Tukey (p=0,05).Paraa análise de AI (n=4), trinta e dois molares receberam cavidades Classe II e foram restaurados com a técnica incremental ou bloco único. Depois de seccionados perpendicularmente, impressões foram realizadas e réplicas em resina epoxi

foram obtidas da superfície interna das restaurações para a análise da formação de fendas através de MEV e os dados foram analisados por ANOVA e teste de Tukey (p=0,05). Para RU (n=8),superfícies de dentina de terceiros molares foram restauradas, seccionadas e armazenadas em água destilada antes do teste de RU. Os dados foram analisados por ANOVA e teste de Tukey (p=0,05). Todas as resinas compostas continham silício, e outros componentes variados. As resinas apresentaram morfologia e tamanhos diferentes de partículas de carga. Filtek Silorane apresentou as menores médias de RS, SR e SO. O armazenamento em água por um ano aumentou a SR para todas as resinas compostas testadas. Filtek Silorane apresentou TP semelhantes aos materiais à base de metacrilato, a menor AI e as maiores médias de RU após um ano de armazenamento. A resina composta de baixa contração Filtek Silorane se mostrou como alternativa aos materiais de formulação convencional, para restauração de dentes posteriores, uma vez que em vários testes realizados ela obteve os melhores resultados.

PALAVRAS-CHAVE: resina composta, caracterização de partículas de carga, sorção, solubilidade, rugosidade, formação de fendas, resistência de união, tensão de polimerização, silorano

ABSTRACT

The aim of this study were: 1) to characterize the chemical elements and the morphology of filler particles in conventional and low-shrinkage composite resins; 2) to evaluate the surface roughness (SR) and 3) the water sorption (WS) and solubility (SO) after finishing/polishing 24h and after 1 year of water storage; 4) polymerization stress (PS) of the composites; 5) To evaluate the internal adaptation (IA) on Class II cavity walls using incremental and bulk filling restorative technique 24h and one year of water storage and 6) the bond strength (BS) of silorane- and methacrylate-based restorative systems after 24h and one year of water storage to dentin. Four composites were evaluated: two low shrinkage (Filtek Silorane and Aelite LS) and two conventional composites (Heliomolar and Tetric N-Ceram). For the BS and IA analysis, the adhesives from the same manufacturer were used: Excite for Tetric N-Ceram and Heliomolar; One-Step Plus for Aelite LS and Silorane System Adhesive for Filtek Silorane. Toidentify the inorganic components and to evaluate the filler particles characteristics, the materials (n=5) were immersed in organic solvents to eliminate the organic phase and observed by SEM and EDX. For the SR evaluation, WS and SO (n=10) their respective finishing and polishing systems were used following the manufacturer instructions. Polished surfaces were analyzed using a profilometer. For the WS and SO, the discs were stored in desiccators until constant mass. Specimens were stored in water and the mass of each specimen was measured. The specimens were dried again and the mass was determined. The WS and SO were calculated from these measurements. Data analyzed by ANOVA and Tukey's test (p=0.05). PS (n=5) was determined by the insertion of the composite between rods of polymethyl methacrylate. Data analyzed by ANOVA and Tukey's test (p=0.05).For IA analysis (n=4), thirty-two molars received Class II cavities and were restoring according to incremental or bulk filling technique. After sectioned perpendicularly, impression were taken and epoxy resin replicas were obtained of the internal surface of restorations to analysis gap formation using scanning electron microscopy (SEM)

and the data was analyze by ANOVA and Tukey's test (p=0.05). For BS test (n=8), dentin surfaces of third molars were bonded, sectioned and stored before BS test. Data was analyzed by ANOVA and Tukey's test (p=0.05). All composite resins contained silicon, but the other components varied. The resins showed different filler particle sizes and morphologies. Filtek Silorane showed the lowest SR, WS and SO means. Water storage for one year increased the WS means for all composite resins tested. Filtek Silorane showed the highest means of bond strength after one year of storage, the lowest formation of gaps and PS similarly methacrylates based materials. The composite resin of low shrinkage Filtek Silorane showed as an alternative to conventional composites for restoration of posterior teeth, since in many tests has obtained the best results.

KEYWORDS: Resin composite, filler characterization, water sorption, solubility, roughness, gap formation, bond strength, polymerization stress, silorane

SUMÁRIO

INTRODUÇÃO

A composição básica das resinas compostas restauradoras é formada pela combinação de diferentes tipos de monômeros como TEGDMA, UDMA e BisGMA, partículas de carga, inibidores, agentes de união (silano) e fotoiniciadores (Calheiros *et al.*,2008; Gonçalves *et al.*,2008). A polimerização das resinas compostas produz contração volumétrica do material de 3 a 5%, que causa danos e problemas na área de união e estruturas adjacentes, dependendo do tipo de cavidade, da quantidade de material aplicado e a forma como o compósito foi inserido (Braga *et al.*,2006; Lee *et al.*,2007; Pfeifer *et al.*,2008).

Na tentativa de reduzir os efeitos da contração de polimerização, técnicas incrementais de inserção de compósitos e diferentes fontes e técnicas de irradiações têm sido propostas (Reis *et al.*,2003; Santos *et al.*,2004; Cunha *et al.*,2009). A modificação na formulação dos materiais restauradores, principalmente na parte monomérica, é outra forma de tentar eliminar ou diminuir a contração de polimerização (Calheiros *et al.*,2008; Gonçalves *et al.*,2008,Pfeifer *et al.*,2008). A formulação de novos compósitos demanda tempo e conhecimento específico da área de polímeros, além disso, a modificação da composição não pode comprometer as propriedades físicas e de manipulação da resina composta.

Uma resina composta específica para dentes posteriores foi desenvolvida com monômeros siloxano e oxirano, ao invés dos tradicionais metacrilatos. A reação de polimerização é diferente e envolve a abertura do anel oxirano, que reduz a contração volumétrica durante a polimerização para menos de 1% (Weinmann *et al.,*2005). Outro material aumentou a quantidade de carga com partículas de vidro e sílica amorfa (84 a 88% por peso e 74 a76% por volume) para reduzir a contração de polimerização (1,4 a 1,9% de contração volumétrica e 0,5 a 0,6% de contração linear) (Boaro *et*

al., 2010). Embora a redução da contração seja clinicamente desejável para o uso dos compósitos restauradores principalmente em dentes posteriores (Schattenberg *et al*.,2007; Rullmann *et al*.,2012), ainda pouco se sabe sobre diferentes tópicos referentes a esses materiais, tais como, a forma de conveniência, sua relação com o adesivo na resistência de união à dentina, a adaptação do material no preparo cavitário, a sorção de água e solubilidade, além da rugosidade superficial após polimento, em função do tipo e tamanho das partículas de carga para cada compósito.

As resinas compostas são classificadas de acordo com o tamanho, conteúdo e tipo da partícula, as quais podem ser de vidro de bário-alumino-silicato, vidro de lítio-boro-bário, trifluoreto de itérbio, quartzo, zircônia e dióxido de sílica. Resinas com partículas maiores que um mícron são chamadas de compósitos de macropartículas, enquanto, as que apresentam partículas menores que um mícron são conhecidas como microparticuladas. Novas classificações incluem as resinas compostas híbridas, microhíbridas e de nanopartículas (Anusavice, 2003; Jung *et al.*, 2007). As partículas podem ter formas irregulares ou serem esféricas dependendo do fabricante. O formato e o tamanho das partículas de carga podem influenciar o polimento, o desgaste dos materiais, além de outras propriedades mecânicas (Reis *et al*., 2003; Takanashi *et al.*,2008).

Desta forma, o estudo *in vitro* teve como objetivo geral avaliar quatro resinas compostas (duas convencionais e duas de baixa contração) quanto às características das partículas de carga, rugosidade superficial, sorção e solubilidade, formação interna de fendas, resistência de união em dentina e tensão de polimerização.

Os objetivos específicos deste estudo foram:

- Caracterizar os tipos das partículas de carga das resinas compostas estudadas, utilizando Microscopia Eletrônica de Varredura (MEV) e Espectometria por Dispersão de Energia de Raio X (EDS) (Artigo 1);
- Avaliar a rugosidade superficial de quatro resinas compostas após procedimentos de acabamento e polimento; e após o armazenamento por 24 horas e 1 ano (Artigo 2);
- Analisar a sorção e a solubilidade dessas resinas compostas, após 1 semana e 1 ano de armazenamento em água (Artigo 2);
- Avaliar a resistência de união à dentina de quatro sistemas restauradores, após 24 horas e 1 ano de armazenamento (Artigo 3);
- Avaliar a adaptação no preparo cavitário das resinas compostas aplicadas em restaurações Classe II, utilizando técnica incremental ou incremento único, em MEV, após 24 horas e 1 ano de armazenamento (Artigo 3);
- Determinar a tensão de contração de polimerização de quatro resinas compostas (Artigo 3).

CAPÍTULO 1

Investigation of filler particle composition and morphology of low-shrinkage and traditional composite resins by SEM and EDX

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ABSTRACT

The purpose of this study was to characterize the chemical elements and morphology of the filler particles in conventional and low-shrinkage composite resins. The main components were identified by energy-dispersive X-ray (EDX) microanalysis, and the filler particles were analyzed morphologically by scanning electron microscopy (SEM). Four composite resins were studied: two conventional resins (Heliomolar and Tetric N-Ceram, Ivoclar Vivadent) and two low-shrinkage composite resins (Aelite LS, Bisco, and Filtek Silorane, 3M ESPE). The materials (five samples of each resin) were immersed in organic solvents to eliminate the organic phase and observed by SEM and EDX. Although the EDX measurements showed a high content of silicon in all of the materials, differences in the elemental composition were identified. The Aelite LS composite resin contained spherical and irregular particles, whereas the other composites contained only irregularly shaped filler particles. The Heliomolar composite had the highest particle size. All composite resins contained silicon, but the other components varied. The resins showed different filler particle sizes and morphologies.

Key Words: filler characterization, silorane, composites, EDX, SEM

INTRODUCTION

The volumetric polymerization shrinkage of regular dental restorative composites after curing is approximately 3–5%. The polymerization shrinkage stress in resin-based materials can cause damage at the resin–tooth interface, formation of marginal gaps, marginal staining, post operative sensitivity, and, consequently, early failure of composite resin restorations (Carvalho et al., 1996; Braga et al., 2006; Lee at al., 2007; Pfeifer et al.,

2008).In an attempt to reduce the effects of polymerization shrinkage, dentists place restorative composites using incremental techniques (Reis et al., 2003; Park et al., 2008, Cunha et al., 2009)and may use “soft-start” polymerization, for which there are three different techniques: stepped, ramped, and pulse-delay (Bouschlicher et. al., 2000a, b; Sahafi et al., 2001).

Changes in the formulation of composite restorative materials have also been made in order to eliminate or reduce volume shrinkage during polymerization (Pfeifer et al., 2008; Calheiros et al., 2008; Gonçalves et al., 2008).The main changes have been in the monomer composition and in the amount of filler particles. New monomers for resin-based composites have been developed and incorporated into commercial composite resins, such as siloranes, dimer acid-based dimethacrylates, tricyclodecane (TCD) urethane, and organically modified ceramics (ormocers) (Ilie et al., 2011).

A reduction in the size of the filler particles incorporated into the monomeric matrix of composite resins seems to have been a tendency over the years (Ferracane et al., 1995).Composites may be classified according to the filler size as nanofills (mean particle size 0.001–0.01 μm), microfills (0.01–0.1 μm), minifills (0.1–1 μm), midifills (1.0–10 μm), and macrofills (10–100 μm) (Bayne et al., 1994; Sherwoodv, 2010);however, most commercial composites are classified as “hybrid” composites, since they contain particles (including silica) in two or more size ranges to improve handling by reducing stickiness (Ilie et al., 2011).With regard to the filler loading, an increase in the content of filler particles (up to 60% by volume, glass and silica) has allowed reduction in the monomer content and, consequently, in the polymerization shrinkage.

The purpose of this study was to investigate whether there are differences in the composition of the filler particles between traditional and low-shrinkage composite resins, using energy-dispersive X-ray (EDX) spectroscopy microanalysis. In addition, the morphological characteristics of the filler particles were determined using scanning electron microscopy (SEM). The research hypotheses tested were that there are differences in the

inorganic composition and in the filler morphology between traditional and low-shrinkage composite resins.

MATERIALS AND METHODS

Two traditional composite resins (Heliomolar and Tetric N-Ceram, Ivoclar Vivadent, Schaan, Liechtenstein) and two low-shrinkage composite resins (AeliteLS, Bisco, Inc., Schaumburg, IL, USA, and Filtek Silorane, 3M ESPE, St. Paul, MN, USA) were selected for this study (Table 1). For each of these materials, five samples were prepared from 60 ± 1 mg of resin. The unpolymerized composite resins were dissolved in 6 mL of acetone (99.5%) and centrifuged for 5 min. This procedure was repeated three times at intervals of 24 h. Chloroform (99.8%) was then used in the same manner (Babbagh et al.; 2004).The filler particles that remained were immersed in 6 mL of absolute ethanol for one day, followed by air-drying overnight at 37° C (Aguiar et al., 2012).The resulting samples were fixed on plastic stubs and sputter-coated with carbon (MED 010, Balzers, Liechtenstein) to eliminate charging effects. The samples were then observed in a scanning electron microscope and analyzed by EDX spectrometry.

EDX analysis was used to detect the main chemical components of the materials. Chemical elements (organic and inorganic) were identified by using a scanning electron microscope equipped with a Vantage EDX system (NORAN Instruments, Middleton, WI, USA). The EDX spectra were acquired for 100 s livetime (voltage 15 kV, dead time 20–25%, working distance 20 mm).

For morphological characterization of the filler particles, samples were observed using a scanning electron microscope (VP 435, Leo, Cambridge, UK). Five repetitions were performed for each composite resin. SEM images of the filler particles were recorded at magnifications of 1000 and 5000 (voltage 15 kV, beam width 25–30 nm, working distance 10–15 mm). Therefore five images at a magnification of 1000 and five at a magnification of

5000 were obtained for each resin to be analyzed. The five images at a magnification of 5000 were used to calculate the size of the filler particles for each composite. The measurement of the sizes was performed using the scale markers on the images.

RESULTS

The chemical elements identified by SEM/EDX analysis are shown in Fig. 1 and Table 2. The inorganic elements found in Heliomolar by EDX were aluminum, fluorine, ytterbium, calcium, and silicon (Fig. 1A). Tetric N-Ceram was found to contain barium, aluminum, ytterbium, zirconium, and silicon (Fig. 1B). Aelite LS contained aluminum and silicon in its inorganic composition (Fig. 1C), and Filtek Silorane contained yttrium and silicon (Fig. 1D). High amounts of silicon were detected in all composites. The two organic elements carbon and oxygen were detected in all materials.

The examination of the filler particles by SEM showed morphological variations among the composite resins. Figures 2–5 show filler particles from Heliomolar, Tetric N-Ceram, Aelite LS, and Filtek Silorane, respectively. The SEM micrographs of the Heliomolar composite resin showed small particles (around 1 μm in size) and many particles larger than 10 μm (Fig. 2). For Tetric N-Ceram (Fig.3) and Filtek Silorane (Fig. 5), most particles were irregularly shaped, with sizes ranging from 0.5 to 1.5 μm; however, the sizes were more homogeneous for Filtek Silorane (the average size was 1 μm). Tetric N-Ceram also showed small spherical particles (Fig. 3B). Irregularly shaped and spherical particles could be observed in Aelite LS (Figs. 4A and B). In this low-shrinkage composite, the size of the irregularly shaped filler particles ranged from 5 to 10 μm and that of the spherical particles ranged from 0.5 to 3 μm.

DISCUSSION

The two low-shrinkage composite resins investigated in this study are designed for using in posterior teeth only. The changes that were made in the

compositions of these composites to reduce polymerization shrinkage compromise their esthetic properties, limiting their indication for anterior teeth. Silorane-based composites polymerize by a cationic ring-opening process, which is different from the polymerization reaction of the traditional methacrylate-based materials. The result of this cationic ring-opening mechanism is a significantly lower volumetric shrinkage (<1%) than in methacrylate-based composites. Whereas in Filtek Silorane the monomer matrix was changed, in Aelite LS the filler particle content was increased to 74% by volume in order to reduce the polymerization shrinkage.

According to its manufacturer, the Aelite LS low-shrinkage composite resin shows 1.4% volumetric shrinkage and 0.5% linear shrinkage. This composite is highly filled and is a hybrid composite, since it contains both irregularly shaped and spherical filler particles with different sizes (ranging from 0.5 μm to 10 μm). The particles are composed of aluminum glass and amorphous silica. In Fig. 4B, it is possible to observe that a higher amount of spherical filler particles has been incorporated into the organic matrix than would be possible for irregular filler particles of the same size. A spherical shape improves the packing of the particles in the matrix (Bayne et al., 1994)and therefore allows an increase in the volume fraction of the filler in the composite (Hosoda et al., 1990; Khan et al., 1992)which tends to reduce the monomer content and, consequently, the polymerization shrinkage (Miyazaki et al., 1991; Baroudi et al., 2007).However, such an increase in the filler volume fraction has a limit, since a high filler loading can lead to a decrease in mechanical properties (Ilie et al., 2011; Kim et al., 2002).

Filtek Silorane contains quartz and yttrium fluoride as filler particles, with a uniform particle size distribution and an average size of 1 μm (Fig. 5B). Quartz is twice as hard as glass and more resistant to dissolution. However, a limitation on its use is that quartz is radiolucent. To overcome this limitation, the manufacturer has added yttrium fluoride as a radiopaque constituent. In this study, both silicon (from quartz) and yttrium were identified by EDX analysis (Sherwoodv, 2010; Abers, 2002).

Although Heliomolar is considered as a microfilled composite, small particles (around 1 μm) and many particles larger than 15 μm were found in this material (Fig. 2) in this study. The EDX analysis detected aluminum, silicon, ytterbium, fluorine, and calcium in the samples of this composite. According to the manufacturer, the filler particles present in Heliomolar consist of silicon dioxide, ytterbium trifluoride, and a prepolymer. The ytterbium trifluoride particles serve as a radiopacifier and a fluoride release agent (Sabbagh et al., 2004; Khan et al., 1992).The prepolymer particles are prepolymerized microfill particles that show the same properties as the matrix. They increase the filler content, and enhance the consistency of the material and its physical properties. Calcium and aluminum may be constituents of these prepolymer particles. The manufacturer does not provide information about the composition of these particles; however, the manufacturer's information suggests that some type of glass containing a calcium fluoroaluminosilicate may form part of the microfiller particles.

Tetric N-Ceram is a hybrid composite resin; most of the filler particles were irregularly shaped, with sizes ranging from 0.5 to 1.5 μm. Small spherical particles were also seen and, according to the manufacturer, these are nanoparticles with a size less than 100 nm. The filler particles represent 56% by volume of the product and consist of barium glass, ytterbium trifluoride, mixed oxides, and prepolymerized filler. Among the chemical elements identified by the EDX analysis, aluminum, barium, and silicon may be constituents of the barium boroaluminosilicate glass filler. Zirconium and silicon are related to the mixed oxide particles. The name of this composite suggests that it includes ceramic fillers such as porcelain, quartz, or zirconia. Fluorine was not detected, possibly because of overlap of the peaks of the elements detected or because of a low concentration of the element.

The elements carbon and oxygen are constituents of the resin monomers used in all of the composite resins. Oxygen in silicon dioxide or colloidal silica particles may be also has been detected. These two elements represent the organic fraction of the composite; some of this organic fraction

may have remained adhering to the filler particles even after organic-solvent dissolution or may have been present under filler particles on the surface of the samples analyzed.

The examination of filler particles by SEM in this study showed morphological variations among the composite resins investigated. The EDX microanalysis also detected differences in the inorganic composition, although the element silicon was always present. In general, the inorganic components were in accordance with the information provided by the manufacturers. The hypotheses that there are differences in the inorganic composition and in the morphological characteristics of the filler particles between traditional and low-shrinkage composite resins were accepted.

ACKNOWLEDGEMENT

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TABLES

Table 1: Materials, manufacturer, composition and batch number of the composites used in this study (Information supplied by the MSDS of the manufacturer).

Material (Manufacturer)	Composition	Batch Number
Heliomolar (IvoclarVivadent, Schaan, Liechtenstein)	Bis-GMA, UDMA, 1,10-decanediol dimethacrylate, camphorquinone, silicon dioxide, ytterbium trifluoride and prepolymerized filler (prepolymers) (46% vol.)	K35053
Tetric N-Ceram (IvoclarVivadent, Schaan, Liechtenstein)	Dimethacrylates, additives, catalysts, stabilizer sand pigments, barium glass, ytterbium trifluoride, mixed oxide and prepolymerized filler (prepolymers) (56% vol.)	L48183
Aelite LS (Bisco Inc., Schaumburg, IL, USA)	Bis-GMA, Bis-EMA, TEGDMA, camphorquinone, glass filler, amorphous sílica (74 vol%)	0900005990
FiltekSilorane (3M ESPE, St. Paul, MN, USA)	Bis-3,4-epoxycyclohexylethyl-phenyl-Methylsilane 3,4 Epoxycyclohexylcyclopolymethylsiloxane, camphorquinone, iodonium salt and electron donor, silanized quartz, yttrium fluoride (55 vol%)	N205711

Abbreviations: bis-phenol Adiglycidylmethacrylate (Bis-GMA), urethane dimethacrylate (UDMA), ethoxylatedbisphenol A dimethacrylate (Bis-EMA) and triethylene glycol dimethacrylate (TEGDMA).

Table 2. The chemical elements identified by EDX analysis of the composites studied.

Composite Resin	Chemical Elements
Heliomolar	O, C, Al, Si, Yb, F, Ca
Tetric N-Ceram	O, C, Al, Si, Ba, Yb, Zr
Aelite LS	O, C, Al, Si
Filtek Silorane	O, C, Si, Y

Abbreviations: O (oxygen), C (carbon), Al (aluminum), Si (silicon), Yb (ytterbium), F (fluorine), Ca (calcium), Zr (zirconium), Ba (barium) and Y (yttrium).

FIGURES

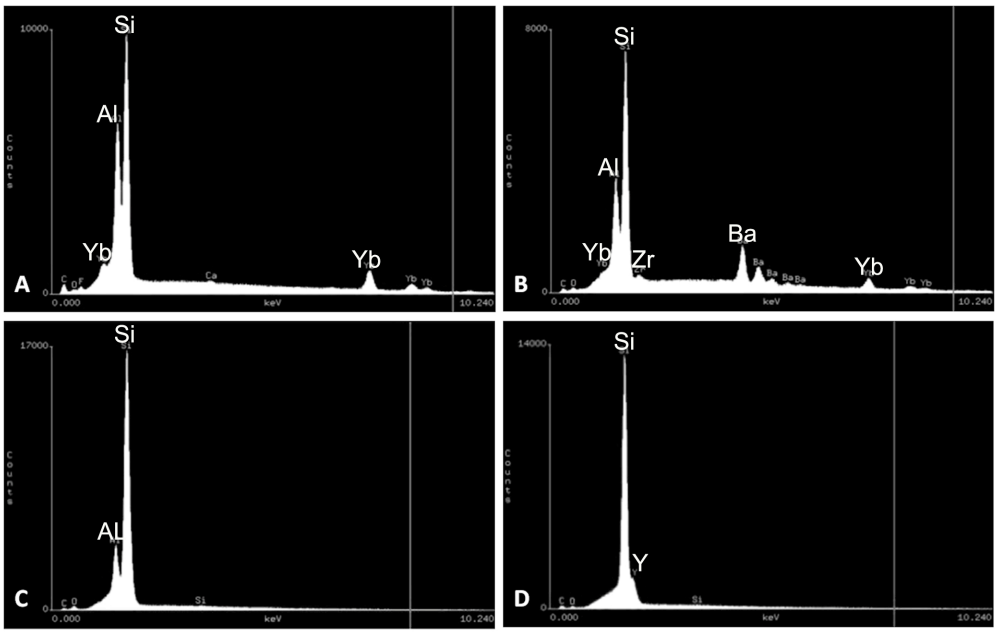

Figure 1. Elements identified by energy dispersive X-ray spectroscopy microanalysis for Heliomolar (A), Tetric N-Ceram (B), Aelite LS (C) and Filtek Silorane (D).

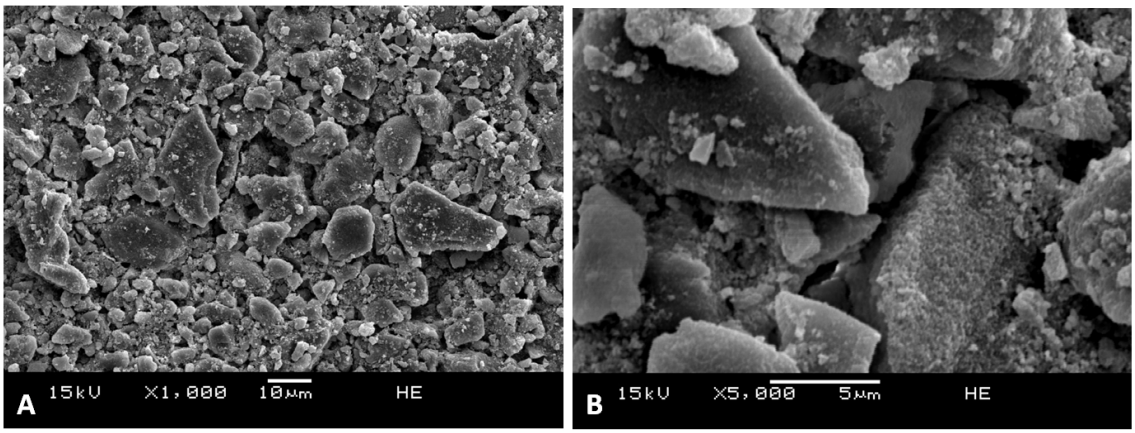

Figure 2. SEM micrograph of Heliomolar resin composite; magnification 1.000X (A) and 5.000X (B).

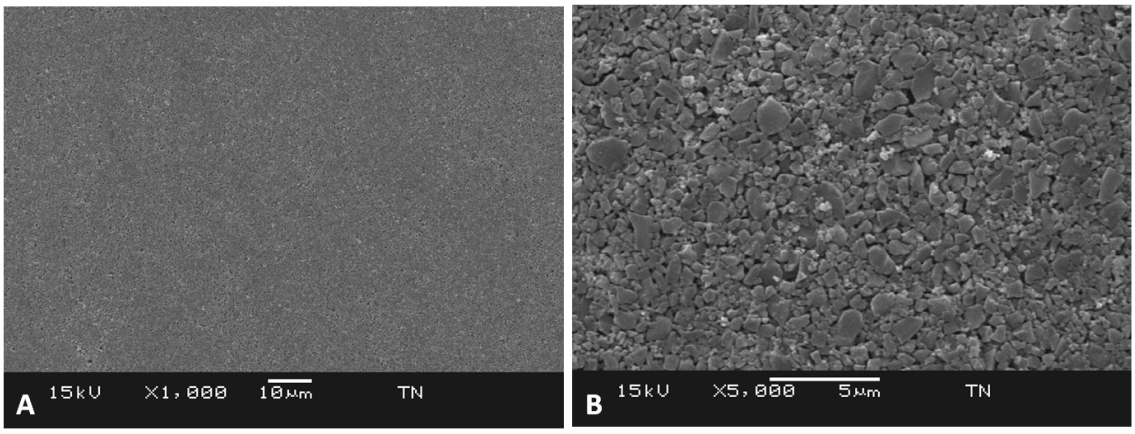

Figure 3. SEM micrograph of Tetric N-Ceram resin composite; magnification 1.000X (A) and 5.000X (B).

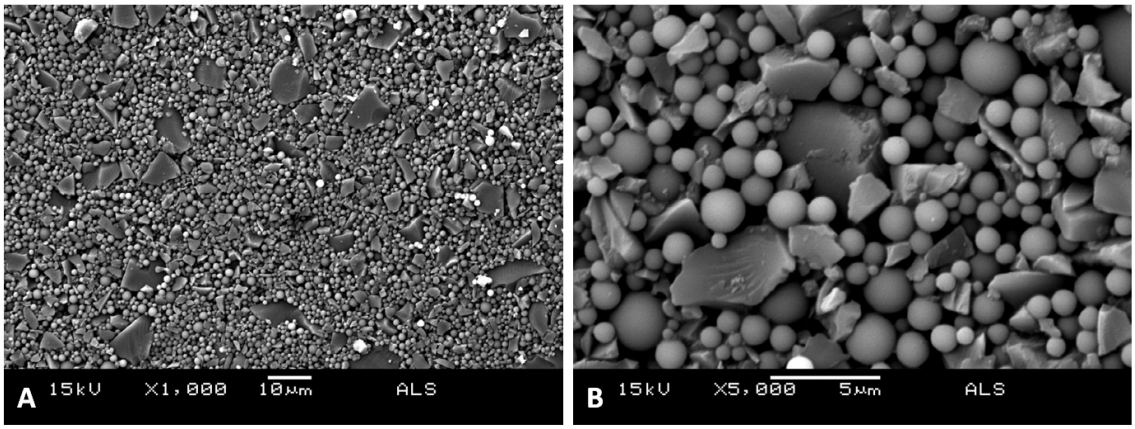

Figure 4. SEM micrograph of Aelite LS resin composite; magnification 1.000X (A) and 5.000X (B).

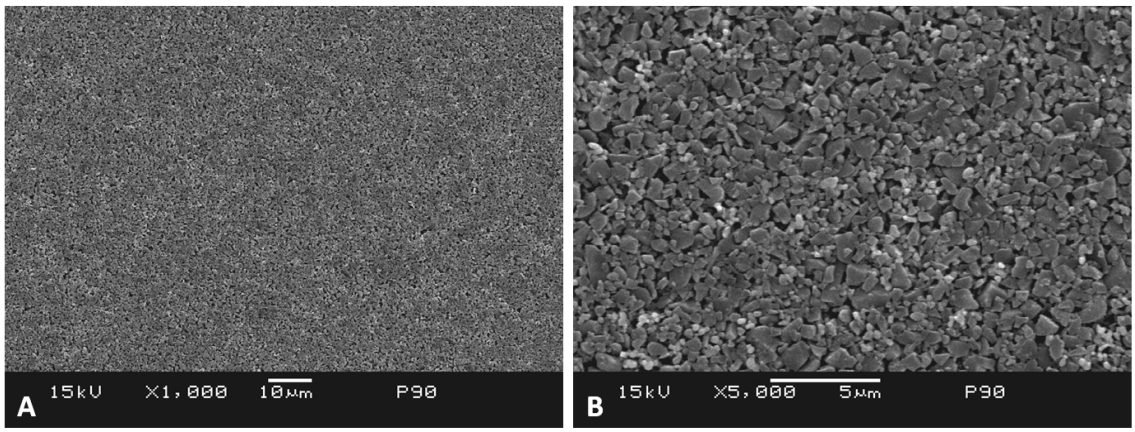

Figure 5. SEM micrograph of Filtek Silorane resin composite; magnification 1.000X (A) and 5.000X (B).

CAPÍTULO 2

Characterization of Water Sorption, Solubility, and Roughness of Silorane- and Methacrylate-Based Composite Resins

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Running Title: water sorption, solubility, and roughness of composite resins

CLINICAL ELEVANCE

This study showed that silorane-based composites demonstrated good performance in all parameters studied (water sorption, solubility, and roughness), suggesting its use as good alternative restorative material.

ABSTRACT

Objective: The objective of this study was to evaluate the surface roughness (SR), water sorption (WS), and solubility (SO) of four composite resins after

finishing/polishing and after one year of water storage. Material and Methods: Two low-shrinkage composites (Filtek Silorane, 3M ESPE and Aelite LS, Bisco Inc.) and two composites of conventional formulations (Heliomolar and Tetric N-Ceram, Ivoclar Vivadent) were tested. Their respective finishing and polishing systems (Sof-Lex Discs, 3M ESPE; Finishing Discs Kit, Bisco Inc., and Astropol F, P, HP, Ivoclar Vivadent) were used following the manufacturer's instructions. Ten disc-shaped specimens of each composite resin were made for each evaluation. Polished surfaces were analyzed using a profilometer after 24 h and one year. For the WS and SO, the discs were stored in desiccators until constant mass was achieved. Specimens were then stored in water for 7 days or one year, at which time the mass of each specimen was measured. The specimens were dried again and dried specimen mass determined. The WS and SO were calculated from these measurements. Data analyzed by two-way ANOVA and Tukey's post-hoc test ($\alpha = 0.05$). Results: Filtek Silorane showed the lowest SR, WS, and SO means. Water storage for one year increased the WS means for all composite resins tested. Conclusions: The silorane-based composite resin results were better than those obtained for methacrylate-based resins. One-year water-storage did not change the SR and SO properties in any of the composite resins.

Keywords: composites resins, water sorption, solubility, roughness, silorane.

INTRODUCTION

Restorative composite resins consist of a polymerizable resin matrix and filler particles that are chemically bonded by silane coupling agents. In recent years, the clinical use of resin composites has considerably increased because of the advances in composite technology and the efficacy of bonding agents.[1-3]

The polymerization shrinkage of composite resins has been reduced, but it remains a concern in the restorative dentistry field. Its effects are related to marginal gap formation by interface debonding, microcracking, or fracture

of the thin surrounding walls of the dental preparations.[4-7] Considerable effort has been invested to minimize shrinkage in order to reduce the stress that stems from polymerization of composite materials. Different strategies are used to reduce polymerization shrinkage: changing the monomeric matrix or increasing the filler load.[8]However, low-shrinkage composites are indicated only for posterior teeth because of the geometry of the class I and II cavity preparations.

Resistance to degradation in the oral environment is essential to the longevity of composite resin restoration.[9] Some properties of composites such as surface roughness (SR), water sorption (WS), and solubility (SO) are important parameters to predict the behavior of composite restorations. WS by composite resins is a diffusion-controlled process that may cause chemical degradation of the material, leading to several drawbacks, such as filler-polymeric matrix debonding[10] and residual monomer release. This process can decrease the mechanical properties of the material[10] and reduce the longevity of resin composite restorations. The SO of resin composites is reflected by the amount of leachable unreacted monomers and filler particle loss.

SR is an important property that affects the appearance of the composite material. A smooth surface improves esthetics and reduces plaque retention, surface discoloration, and tissue inflammation.[11-15] It also increases the patient's comfort.[16] SR, WS, and SO properties depend on the formulation of each material, including filler content, size, shape, and interparticle spacing, the monomer type, degree of curing, and the efficiency of the filler-matrix bonding.[14,17]

The aim of this study was to investigate the surface roughness (SR), water sorption (WS), and solubility (SO) of two low-shrinkage composites (Filtek Silorane and Aelite LS) and two regular composites (Heliomolar and Tetric N-Ceram) after finishing and polishing and after one year of water storage. The null hypothesis tested was that low-shrinkage composites would

not present the same properties investigated (SR, WS, and SO) when compared to conventional restorative resin-based materials.

MATERIAL AND METHODS

Four commercial composite resins were selected: two low-shrinkage composites: Filtek Silorane (3M ESPE, St. Paul, MN, USA) and Aelite LS (Bisco, Inc., Schaumburg, IL, USA); and two regular composites: Heliomolar and Tetric N-Ceram (Ivoclar Vivadent, Schaan, Liechtenstein). The respective finishing and polishing systems were also used: Sof-Lex Finishing and Polishing Discs (3M ESPE), Finishing Discs Kit (Bisco Inc.), and Astropol F, P, AH (Ivoclar Vivadent). The compositions and lot numbers of the composite resins and the finishing and polishing systems are listed in Tables 1 and 2, respectively.

1. Surface Roughness and SEM Observations

For evaluating the SR, eighty cylindrical specimens of composite resins (n = 10) 2.0 mm thick and 6.0 mm in diameter were prepared in Teflon molds. The composites were inserted into the mold incrementally using a composite placement instrument (Suprafill Plastic Filling, Duflex SS White, Juiz de Fora, MG, Brazil). Two layers were enough to fill the cavity and each increment was cured for 40 seconds with a visible light-curing unit, Demetron Optilux 501 (Kerr Corp., Orange, CA, USA). The second layer was covered with a polyester mylar strip (Dentsply, Petrópolis, RJ, Brazil), pressed with a glass slide (Glasstécnica Import. Com. de Vidros Ltda., São Paulo, SP, Brazil) and light-cured.[1,18]All specimens were stored in distilled water at 37°C for 24 h.

The specimens were finished and polished by a single investigator and the cups and disc polishers were applied using a low-speed hand-piece (Intramatic ES, Kavo do Brazil, Joinville, SC, Brazil) for 15 s at a speed of 10,000 rpm. The sequence and time of application of the finishing systems are described in Table 2.

Half of the specimens of each composite were stored in water for 24 h at 37°C (baseline SR) and the other half were stored in 10 mL of distilled water in eppendorfs for one year before testing. To measure the SR of the specimens, a profilometer (Surfcorder SE 1700, Kosaka Laboratory Ltda, Tokyo, Japan) with a speed of 0.05 mm/s (0.25 mm cut-off) was used. Three measurements taken in different directions were recorded for all specimens to obtain the SR average (Ra) for each specimen. Two-way analysis of variance (ANOVA) was used to evaluate the data from the profilometric experiment. To identify significant differences, a Tukey's test at a 5% level of significance was used (MINITAB 15, State College, PA, USA).

Three specimens per group were randomly assigned for observation using scanning electron microscopy (SEM) (JSM 5600, Jeol Inc., Peabody, MA, USA). Specimens were sputter-coated with gold to a thickness of approximately 50 Å in a vacuum evaporator (SCD 050, Bal-Tec AG, Balzers, Liechtenstein) and photomicrographs of a representative area of the surfaces were taken at 200x.

2. Water Sorption and Solubility

The WS and SO analyses were assessed following short- (7 days) and long-term (one year) immersion periods (n = 10). The specimens of each composite resin were prepared using Teflon molds (2.0 mm in thickness and 6.0 mm in diameter). After filling the mold, the composite resin surface was covered with a polyester strip (Dentsply, Petrópolis, RJ, Brazil) and glass slide (Glasstécnica Import Com. de Vidros Ltda, São Paulo, SP, Brazil). The resulting samples were then compressed to avoid porosity and to remove the excess. Specimens were light-cured from the surface with a halogen light-curing unit (Optilux 501; Demetron/Kerr Corp., Orange, CA, USA).

The resin discs were stored in a desiccator (Pyrex, São Paulo, SP, Brazil) at 37°C for 22 h until constant mass was achieved (m1). The masses of these completely dried specimens were recorded (Chyo Balance JK 180; Chyo Corp., Tokyo, Japan). Specimens were then stored for 7 days or one

year in water at 37°C, and the water-saturated mass was measured (m2). Finally, the specimens were dried again in the desiccator until obtaining constant mass and their masses were once again determined (m3). The difference in mass between the initial dry and final dry mass represented the amount of SO (m1 − m3/volume of specimen), which was analyzed by two-way ANOVA and Tukey's post-hoc test ($\alpha = 0.05$). The difference in mass between the saturated and final dry specimens provided WS values (m2 − m3/volume of specimen), which were analyzed by two-way ANOVA and Tukey's post-hoc test ($\alpha = 0.05$).

RESULTS

1. Surface Roughness and SEM Observations

Two-way ANOVA indicated that the factor "composite resin" ($p < 0.0001$) significantly influenced SR results. Conversely, the statistical analysis revealed no significant differences for the factor "evaluation time" ($p = 0.167$) or for interaction between factors ($p = 0.223$). A summary of the SR means for the composite resins is shown in Table 3. Analysis of data with respect to differences in composite resins showed the lowest SR means for Filtek Silorane ($p < 0.05$). Heliomolar, Tetric N-Ceram, and Aelite LS did not differ significantly among themselves ($p > 0.05$). The evaluation time did not influence the SR results ($p > 0.05$).

Representative photomicrographs of the polished specimens are shown in Figures 1–4. After storage for one year, some porosity could be observed on the surfaces of the Aelite LS (Fig. 1) and Heliomolar (Fig. 2) composite resins. Nevertheless, Filtek Silorane (Fig. 3) and Tetric N-Ceram (Fig. 4) showed the smoothest surfaces with similar characteristics before and after water storage for one year.

2. Water Sorption and Solubitily

Two-way ANOVA indicated that the factors “composite resin” ($p = 0.023$) and “evaluation time” ($p = 0.030$) significantly influenced results. No interaction between factors ($p = 0.165$) was identified. A summary of the WS means for the composite resins is shown in Table 4. Analysis of the data with respect to differences in composite resins showed the lowest WS means for Filtek Silorane ($p < 0.05$). The composites differed significantly among themselves ($p > 0.05$). Heliomolar presented the highest WS ($p > 0.05$). The WS of all composites increased after the storage of specimens for one year in water ($p > 0.05$).

A summary of the SO means for the composite resins is shown in Table 4. Two-way ANOVA indicated that the factor “composite resin” ($p = 0.001$) significantly influenced SO results. Conversely, the statistical analysis revealed no significant differences for the factor “evaluation time” ($p = 0.114$) or for interaction between factors ($p = 0.175$). Analysis of the data with respect to differences in composite resins showed the lowest SO means also for Filtek Silorane ($p < 0.05$). The evaluation time did not influence the SO results ($p > 0.05$). Tetric N-Ceram yielded significantly lower SO means than did Aelite LS ($p < 0.05$). The storage time did not influence the SO results ($p > 0.05$).

DISCUSSION

An important factor in determining the SR is the intrinsic roughness of a composite material, which is determined by the size, shape, and quantity of the filler particles.[19] Filtek Silorane contains quartz and yttrium fluoride as filler particles. Its average of particle size is 0.1–2 μm and the filler volume is 55% according to the manufacturer´s information. The lowest SR was observed for Filtek Silorane, whereas other materials did not differ among themselves. Also, the surfaces analyzed by SEM did not change after storage for one year. The WS and SO means were also the lowest for Filtek Silorane, which

contains silorane monomers and is a unique material that does not contain methacrylated monomers.

The finishing and polishing systems influence the SR, the gloss, and the maintainance of the color of restorations.[20-22] Most of the published data shows that existing polishing systems provide sufficiently smooth surfaces, with Ra values ranging from 0.02 μm to 0.56 μm.[20,23,24] All means obtained in this study for the Filtek Silorane, Heliomolar, Tetric N-Ceram, and Aelite LS composites are included in this range. Other studies have shown that when the finishing/polishing system and composite material is from the same manufacturer, their compatibility and polishing results are significantly better.[25-29] Polishing particles must be harder than the filler particles to ensure that the removal of the resin matrix and the fillers would be accomplished in the same way during the polishing.[25,27,29] Sof-Lex discs are composed of aluminum oxide, which is able to cut the filler particles and the polymerized matrix in almost the same way, which explains the lower values of SR and the smooth surface for Filtek Silorane.

The photomicrographs showed that initially the surfaces after finishing and polishing were smooth, but after a year of water storage, the surface Heliomolar and Aelite LS showed some irregularities and porosity. Heliomolar is the lowest filler-loaded (46% by volume), whereas Aelite LS presents the highest filler content (74%). Conversely, Heliomolar has a higher monomeric content than Aelite LS. One of the reasons for the change in SR for Heliomolar is that the water-exposed polymerized organic matrix may be degradated or dissolved, although no alterations were observed for SO analysis. On the other hand, the finishing and polishing for Aelite is more difficult due to the high amount of filler particles, which can be seen in the exposed composite surface after storage for one year (Fig. 1B).

The ISO 4049 standard established that the maximum WS value is 40 $\mu g/mm^3$, whereas the maximum SO value is <7.5 $\mu g/mm^3$. No composite exceeded the maximum WS value, even after one year stored in water, at which point the means increased significantly. The WS for the composite

resins tested in this study ranged from 9.3 to 28.1 μg/mm^3. For SO, the Filtek Silorane and Tetric N-Ceram showed lower means than those stablished by ISO 4049. The SO for the composite resins tested in this study ranged from −1.6 to 12.2 μg/mm^3.

Toledano et al.[30] (2003)reported that WS and SO mainly depends on the resin compositions. The silorane-based composite Filtek Silorane had statistically the lowest values of WS and SO. Silorane is a monomer, with a combination of hydrophobic siloxane and low-shrinkage ring-opening oxirane.[8] Its cationic photo-initiated polymerization reduce the polymerization shrinkage and increasing the degree of conversion.[8,31,32] Thus, the WS of Filtek Silorane is expected to be low. These findings are in accordance with the literature, which report that silorane-based composites exhibit greater hydrophobic behavior than methacrylate-based composite resins.[33,34]

Some hydrophilic constituents such as urethane dimethacrylate (UDMA) or resin molecules that contain hydrophilic moieties increase WS,[17] as observed in Heliomolar, which showed the highest WS means. Tetric N-Ceran showed more WS than Aelite LS, which is a low-shrinkage composite. Although the Tetric N-Ceran and Aelite LS composites contain the same momoneric composition (Bis-GMA, Bis-EMA, and TEGDMA resins), there is a significant difference when the filler particle content was compared (56% and 74% by volume, respectively). The high volume of filler and consequently low monomeric content resulted in reduction of WS, because the WS is mainly promoted by the organic matrix.[35]

Some factors affect the SO of composites, such as the number and the size of leachable species, the type of monomers, the quality of resin–filler adhesion, the solvents, immersion time, and temperature.[30,35] The mass of the components eluted from the composite is found through the water SO data.

The siloxane compound presented in the Filtek Silorane sample clearly provided a material with much lower SO than the other samples tested in this study[37].This confirmed previous findings, which have indicated that this

material is stable in aqueous environments.[34-37] The high filler-loaded Aelite LS composite presented the highest mean of SO after one year. It is possible that the water that is in contact with a silica surface perhaps breaks the siloxane bonds, forming silanol groups, which facilitates particle debonding. Because the hydrolytic stability of coupling agents can vary according to the type of filler particles,[38] no conclusive evidence can be provided to indicate that hydrolytic degradation of the fillers affects the SO behavior of dental composites.[39]

In this study, the length of storage time only influenced the WS, which was higher after one year than the baseline measurements. For the SO, the storage time did not change the values, although the means tended to increase after long-term water storage. Several investigations have analyzed the WS and SO of resin-based materials; however, it is difficult to compare the data because the studies have used different storage periods, expression units[35] and sample dimensions.[30] Regarding the properties studied, silorane-based composites are shown to be a better alternative for low-shrinkage restorative materials as compared to increasing the filler content of composite resins for the purpose of decreasing the polymerization shrinkage.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions can be drawn:

1. The Filtek Silorane, silorane-based material showed lower SR, WS, and SO results than those obtained for methacrylate resin-based materials. Thus, the null hypothesis was partially rejected.
2. The storage in water for one year increased the WS for all composite resins tested, while no changes was observed for SO.

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TABLES

Table 1 Composition of the composite resins tested in this study.

Material (Manufacturer)	Resin monomer	Filler type	Filler volume (%)	Particle size (μm)	Batch number
Filtek Silorane (3M ESPE, St. Paul, MN, USA)	Silorane	silanized quartz, yttrium fluoride	55%	0.1–2 μm	N205711
Aelite LS (Bisco, Inc., Schaumburg, IL, USA)	Bis-GMA, Bis-EMA, TEGDMA	glass filler, amorphous sílica	74%	~1,1 μm	0900005990
Heliomolar (Ivoclar Vivadent, Schaan, Liechtenstein)	Bis-GMA, UDMA	silicon dioxide, ytterbium trifluoride	46%	0.04–0.2 μm	K35053
Tetric N-Ceram (Ivoclar Vivadent, Schaan, Liechtenstein)	Bis-GMA, Bis-EMA, TEGDMA	barium glass, ytterbium trifluoride	56%	0.04–3 μm	L48183

Silorane: Bis-3,4-Epoxycyclohexylethyl-Phenyl-Methylsilane, 3,4-Epoxycyclohexylcyclopolymethylsiloxane silanized; Bis-GMA: Bis-phenol A diglycidylmethacrylate; Bis-EMA: Ethoxylated bisphenol A dimethacrylate; UDMA: Urethane dimethacrylate; TEGDMA: Triethylene glycol-dimethacrylate

Table 2 Composition, mode of application, and times of finishing and polishing systems used in this study.

Finishing and polishing material	Type	Composition	Application	Batch number
Sof-Lex (3M ESPE, St. Paul, MN, USA)	Discs	Coarse, medium fine, superfine aluminium oxide disc	From dark/coarse to light/superfine with a mandrel to polish the surfaces, 15 s for each disc	P050406
Finishing Disc Kit (Bisco, Inc., Schaumburg, IL USA)	Discs	Coarse (brown), medium (green), fine (blue), ultrafine (tan) aluminum oxide disc	Use the sequence: coarse (brown), medium (green), fine (blue), ultrafine (tan) with a mandrel to polish the surfaces, 15 s for each disc	N-71000P
Astropol F, P, HP (Ivoclar Vivadent, Schaan, Liechtenstein)	Cups	Silicone carbide, aluminum oxide, diamond	Finishing with Astropol F (gray), polishing with Astropol P (green), high-gloss polishing with Astropol HP (dusky pink), 15 s for each cup	J16078, J16079, J15646

Table 3 Mean SR produced by the finishing instruments initially and after one year of water storage.

Composite resins/finishing systems	Baseline (μm)	One Year (μm)
Filtek Silorane/Sof-Lex Discs	0.15 ± 0.01 Aa	0.17 ± 0.05 Aa
Aelite LS/Finishing Disc Kit	0.28 ± 0.02 Ba	0.24 ± 0.06 Ba
Heliomolar/Astropol F, P, HP	0.20 ± 0.02 Ba	0.27 ± 0.09 Ba
Tetric N-Ceram/Astropol F, P, HP	0.25 ± 0.04 Ba	0.27 ± 0.10 Ba

Groups with different uppercase (column: comparison among composite/polishing agent within the same evaluation time) and lowercase letters (row: comparison among the evaluation time within the same composite/polishing agent) are significantly different.

Table 4 Mean WS and SO for each sample group

Composite	Sorption (μg/mm^3)		Solubility (μg/mm^3)	
	Baseline	One Year	Baseline	One Year
Filtek Silorane	9.3 ± 1.9 Da	12.4 ± 0.6 Db	-1.6 ± 0.7 Ca	-1.7 ± 0.5 Ca
Aelite LS	17.6 ± 2.0 Ca	18.1 ± 1.6 Cb	10.6 ± 2.1 Aa	12.2 ± 0.8 Aa
Heliomolar	22.3 ± 3.4 Aa	28.1 ± 1.7 Ab	7.6 ± 1.1 ABa	10.5 ± 0.5 ABa
Tetric N Ceram	19.2 ± 1.9 Ba	24.1 ± 1.0 Bb	4.7 ± 1.3 Ba	6.1± 0.5 Ba

Groups with different uppercase (column: comparison among composite within the same evaluation time) and lowercase letters (row: comparison among the evaluation time within the same composite) are significantly different.

FIGURES:

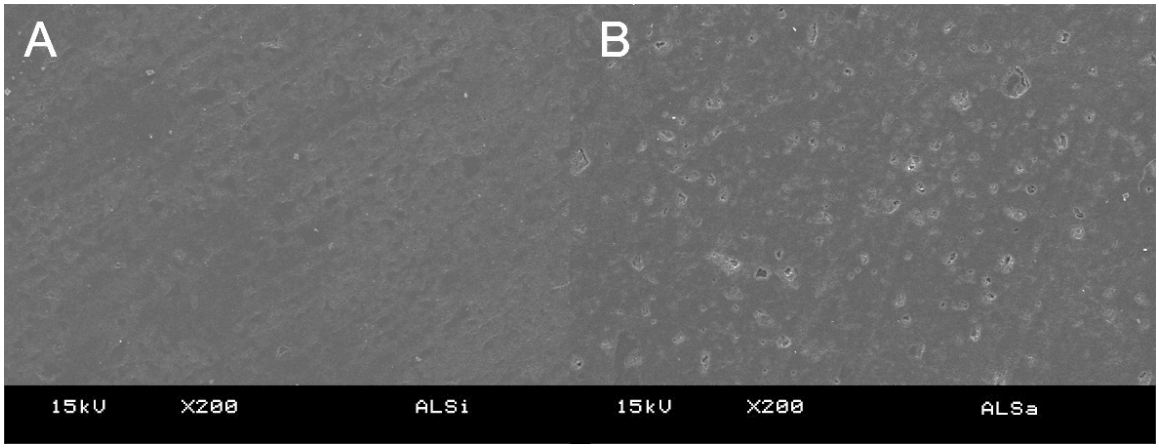

Figure 1. SEM photograph of Aelite LSlow-shrinkage composite resin surface after finishing and polishing with Finishing Disc Kit (A)and after one-year storage in water (B) (magnification 200x).

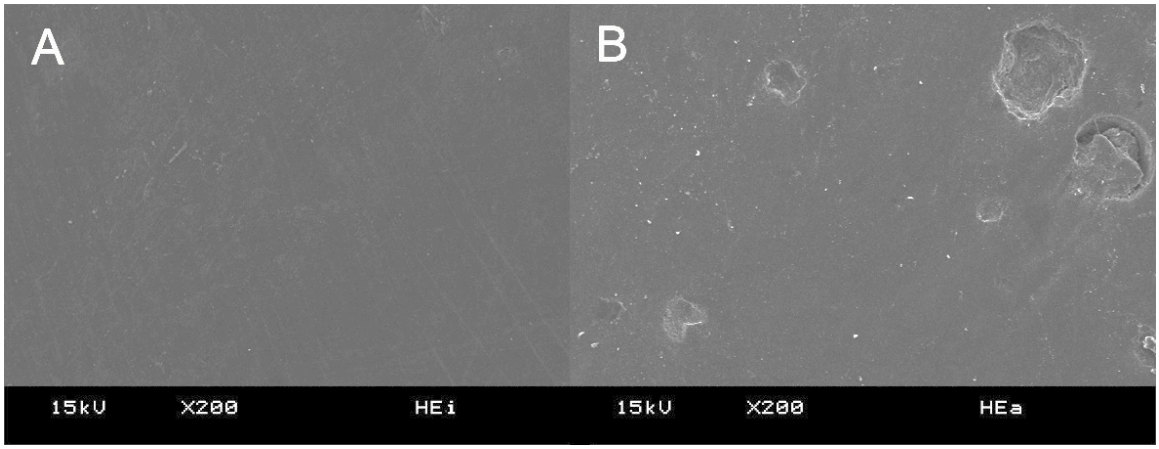

Figure 2. SEM photograph of Heliomolar composite resin surface after finishing and polishing with Astropol F, P, HP (A)and after one-year storage in water (B) (magnification 200x).

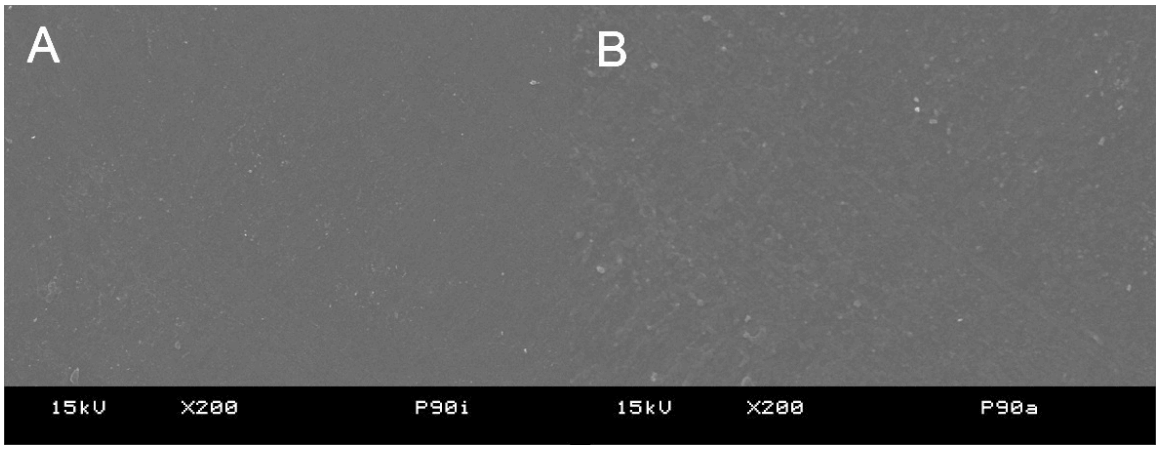

Figure 3. SEM photograph of Filtek Silorane low shrinkage composite resin surface after finishing and polishing with Sof-Lex Discs (A)and after one-year storage in water (B) (magnification 200x).

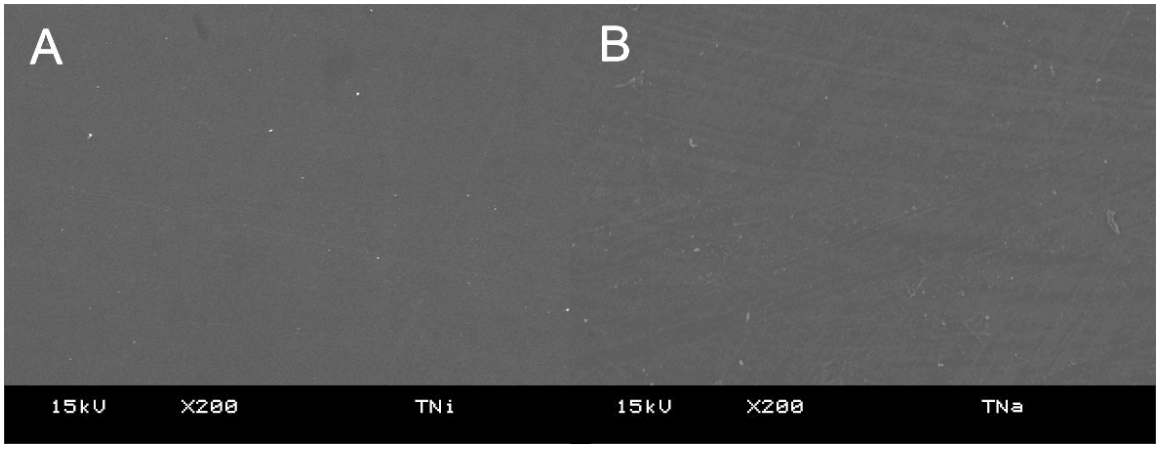

Figure 4. SEM photograph of Tetric N-Ceram composite resin surface after finishing and polishing with Astropol F, P, HP(A)and after one-year storage in water (B) (magnification 200x).

CAPÍTULO 3

Internal adaptation analysis, bond strength, and polymerization stress of silorane- and methacrylate-based composites

Short title: Internal fit, bond strength, and polymerization stress of resin composites.

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Keywords: resin composites, silorane, bond strength, gap formation, polymerization stress

ABSTRACT

Objectives: To evaluate the internal adaptation, bond strength, and polymerization stress of silorane- and methacrylate-based composite. **Methods:** Three methacrylate-based composites (Heliomolar®; Tetric N-Ceram® and Aelite™ LS) and one silorane-based composite resin (Filtek™ Silorane) were tested. Polymerization stress (n = 5) was determined by the insertion of the composite between rods of polymethyl methacrylate. The ratio of the maximum force of contraction was recorded and the cross-sectional area of the rod was used to calculate the nominal stress. Bond strength was evaluated by microtensile bond test. Dentin surfaces of human third molars were bonded, sectioned, and stored for 24 h or 1 year in distilled water before the bond strength test. The ratio of maximum force and the adhered area was used for the bond strength calculation. For internal adaptation analysis, third molars received Class II cavities and were restored according to either an incremental oblique or bulk-filling technique. After being sectioned perpendicularly, impressions were taken and epoxy resin replicas were obtained of the internal surfaces of the restorations (after 24 h and 1 year of storage) to analyze gap formation using scanning electron microscopy. **Results:** Filtek™ Silorane showed the highest mean bond strength after one year of storage, the lowest formation of gaps, and polymerization stress similar to methacrylate-based materials. **Conclusions:** Results suggest that silorane restorative material yielded high and durable dentin bond strength, less gap formation, and polymerization stress comparable to that of regular composites, thus showing it to be a good alternative to traditional composite resins.

INTRODUCTION

The basic composition of a resin composite is formed from different types of monomers such as triethylene glycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA), and bis-phenol A diglycidyl methacrylate (BisGMA),

filler, inhibitors, silane couple agent and photoinitiators.[1,2] The polymerization of composite resins produces shrinkage of the restorative material of approximately 3–5% by volume, which causes damage and problems at the bonded interface, depending on the type of cavity preparation, the volume of material polymerized, and how this composite was placed.[3-5]

In an attempt to reduce or eliminate the effects of polymerization shrinkage, techniques for incremental placement,[6,7] different light-curing sources, irradiation techniques,[8] and the use of low-shrinkage composites or a low-modulus intermediate flowable layer, known as the elastic wall concept,[9] have been proposed. Low-shrinkage composites present new monomeric formulation or higher filler loading that decreases the organic content of commercial composites.[1,2,5] The formulation of new composites requires time and expertise in the field of polymers; additions and composition changes must not compromise the physical properties and handling of the composite.

A specific posterior composite was developed based on the monomers siloxane and oxirane (Filtek Silorane, 3M ESPE, St. Paul, MN, USA) instead of traditional methacrylates. The polymerization reaction for these compounds is different from that of methacrylates and involves the opening of the oxirane ring.[10]Because the resin matrix of the silorane composite significantly differs from that of conventional methacrylate-based composites, a new bonding agent needed to be used with the silorane composite. Filtek Silorane is therefore presented with a two-step self-etch primer, called Silorane System Adhesive (3M ESPE). This adhesive has features of conventional methacrylate adhesives, especially in regard to its bonding mechanism to dentin. An adaptation was needed to make it compatible with the highly hydrophobic silorane matrix.[11] Another material, Aelite LS (Bisco, Inc., Schaumburg, IL USA), increased the amount of filler particles with glass and amorphous silica (84–88% by weight and 74–76% by volume) to reduce the polymerization shrinkage (1.4–1.9% volumetric shrinkage and 0.5–0.6% linear shrinkage)

Although the polymerization shrinkage stress reduction is clinically desirable, especially with posterior teeth resin composites,[12] little is known

relative to these materials, such as the shape of convenience, the bond strength of composites using the specific bonding agents, and the adaptation of the restorative material in the internal cavity walls using Class II cavities. The aim of this study was to evaluate and compare the polymerization stress, the internal adaptation on cavities walls using different restorative techniques (incremental versus bulk filling) in Class II cavities, and the bond strength of silorane- and methacrylate-based restorative systems to dentin after 24 h and 1 year of water storage. The null hypothesis tests were: (1) that there is no difference in polymerization stress between silorane-based and traditional composites; (2) the type of restorative system and aging time had no influence on internal adaptation; and (3) there is no difference in bond strength of adhesives to dentin when compared to the baseline values and after long-term storage.

MATERIAL AND METHODS

Four commercial composite resins (shade A2) were evaluated: two low-shrinkage composites (Filtek™ Silorane, 3M ESPE, St. Paul, MN, USA and Aelite™ LS, Bisco, Inc., Schaumburg, IL, USA) and two regular composites (Heliomolar® and Tetric N-Ceram®, Ivoclar Vivadent, Schaan, Liechtenstein) (Table 1). For the bond strength test and internal adaptation analysis, three adhesive system from the same manufacturer at the same composite resins were used: ExciTE® (Ivoclar Vivadent, Schaan, Liechtenstein) for Tetric N-Ceram and Heliomolar; One-Step® Plus (Bisco, Inc., Schaumburg, IL, USA) for Aelite LS, and Silorane System Adhesive for Filtek Silorane.

Polymerization stress measurements:

Polymerization stress was measured using rods of polymethyl methacrylate (PMMA) as the bonding substrate for the composite, with diameters of 5 mm (n=10). The rods were sectioned in 13 and 28 mm segments. For the 13 mm rods, one of the flat surfaces was lapped and polished by hand using #600–1200 sandpaper and felt disks with 1 μm alumina paste (Alumina 3, ATM,

Altenkirchen, Germany) to allow for light transmission during photoactivation. One of the flat surfaces of the 28 mm rods was sandblasted with alumina (250 μm) for 10 s at a distance of 1 cmand treated with a methyl methacrylate monomer (JET Acrílico Auto Polimerizante, Artigos Odontológicos Clássico, São Paulo, SP, Brazil). These surfaces received two thin layers of an unfilled resin (Scotchbond™ Multipurpose Plus, bottle 3, 3M ESPE, St. Paul, MN, USA), light-activated with 12 J/cm^2 (600 mW/cm^2 for 30 s). Except for the Filtek Silorane, was applied a thin layer of the Adhesive of the Silorane System Adhesive (3M ESPE, St. Paul, MN, USA).

The rods were attached to the opposite fixtures of a universal testing machine (Instron 5565, Canton, MA, USA). On the lower fixture, the 13 mm rod was fixed to a stainless steel attachment with a slot, allowing positioning of the light guide in contact with its polished surface. The 28 mm rod was attached to the upper fixture, which was connected to the load cell. The distance between the rods was 1 mm (cavity configuration factor C = 2.5; volume = 29 mm^3). After the insertion of the composite, an extensometer (model 2630-101, Instron, Canton, MA, USA) was attached to the rods in order to monitor the distance between them during the test and provide feedback to the machine's actuator to re-establish the initial distance. Therefore, the value registered by the load cell corresponded to the force necessary to maintain the initial height of the specimen in opposition to the force exerted by the polymerization shrinkage of the composite. Light-activation was carried out using a quartz–tungsten–halogen light-curing unit (VIP Junior, BISCO, Schaumburg, IL, USA). After propagating through the length of the 13 mm rod, the irradiance reaching the composite surface was 570 mW/cm^2. The irradiance was periodically checked with a dental radiometer (model 100, Kerr Demetron Corp., Orange, CA, USA). A 20 s exposure for tradicional composites and 40 s for Filtek Silorane was used, providing a radiant exposure of approximately 18 J/cm^2. The contraction force was monitored for 5 min from the onset of photoactivation and the maximum nominal polymerization stress (in MPa) was calculated by dividing the maximum force value by the cross-sectional area of the rod. Statistical analysis was

performed with statistical software (MINITAB 15, State College, PA, USA). One-way analysis of variance (ANOVA) for the restorative systems factor was performed, followed by a Tukey's post hoc test ($p < 0.05$).

Internal adaptation analysis:

Thirty-two freshly extracted caries-free human third molars were selected for this part of the study and stored in a solution of distilled water with thymol 0,2% at 4°C for up to 1 month after extractions. Teeth were obtained and used in accordance with a protocol approved by the Ethics Committee in Research of the School of Dentistry of Piracicaba, State University of Campinas. The cusps were abraded using a wet-ground #320-grit silicon carbide paper and then polished using #600-grit. Standardized Class II preparations of the mesial surface of the teeth were made using 3145 diamond burs (KG Sorensen, Cotia, SP, Brazil) with a high-speed hand piece (Turbina Extra Torque 605, Kavo do Brasil,Joinville, SC, Brazil) under water refrigeration (n = 4).

Preparation dimensions were as follows: buccolingual width: 4.0 mm; gingivo-occlusal width: 5.0 mm; axial wall: 2.5 mm depth. The occlusal margins were located in the enamel and the gingival margin was located in the dentin. Burs were replaced after three preparations. Cavity preparation was completed with 3145FF (extra-fine) diamond burs (KG Sorensen, Cotia, SP, Brazil) under water refrigeration. The resulting preparations were randomly distributed into 8 groups (n = 4) according to the restorative system (Heliomolar/ExciTE, Tetric N-Ceram/ExciTE, Aelite LS/One-Step Plus, Filtek Silorane/Silorane Adhesive System) and the filling technique (incremental oblique and bulk-filling techniques). The adhesive systems were applied following the manufacturer's instructions (Table 2).

To restore the Class II cavity, Omni-Matrix™ (Ultradent Products Inc., South Jordan, UT, USA) was used. For the incremental oblique technique group, the resin composites were applied in four increments (±2.0 mm thick each increment) and individually light-activated (VIP Junior, BISCO, Schaumburg, IL,

USA) under irradiance of 600 mW/cm^2, which was constantly monitored with a radiometer, with the distal end of the light-curing tip positioned perpendicular to the occlusal surface of the cavity (Reis et al. 2003). For the bulk-filling group, the cavity was filled in a single increment and subjected to light-activated curing for 40 s. The restored teeth were then stored at 37°C in distilled water for 24 h.

Each restoration was mesio-distally cross-sectioned with a diamond blade, obtaining two half parts. Both halves were polished with #600-, #1200-, and #2000-grit SiC papers, followed by diamond pastes (3-, ½-, and ¼-grit) and placed in an ultrasonic cleaner (Thornton USC 1400, Unique Group, Indaiatuba, SP, Brazil) for 10 min to remove the polishing debris.

The same prepared restorations were evaluated 24 h and 1 year after polishing. For the 1-year analysis, the restorations were stored in distilled water at 37°C in a light-free environment. Impressions of the polished surfaces were taken with a low-viscosity polyvinyl siloxane material (Express™ XT, 3M ESPE, St. Paul, MN, USA) and the impressions were poured with epoxy resin (Buehler Ltd., Lake Buff, IL, USA). Afterwards, the replicas were gold-sputter-coated (Balzers-SCD 050 Sputter Coater, Balzers, Liechtenstein) and observed using SEM (JEOL, JSM-5600LV, Tokyo, Japan) for the evaluation, measurement, and classification of the cavity margins with 200× magnification. For each specimen, it was necessary to take approximately 12 images in order to scan the entire perimeter of the restoration. For the measurement of marginal gaps, the Image J software (National Institute of Health, Bethesda, MD, USA) was calibrated based on the scale bar present in the SEM images. This was possible because all the photos were taken at the same magnification (200×). Then the entire perimeter of the cavity was measured (in mm) to enable the calculation of the percentage of gaps. The gaps were measured and the value converted to a percentage based on the perimeter of each specimen. Statistical analysis was performed with statistical software (MINITAB 15, State College, PA, USA). Three-way analysis of variance (ANOVA) for restorative systems, restorative placement technique, and storage time factors was performed, followed by a Tukey's post hoc test ($p < 0.05$).

Bond strength test:

Thirty-two freshly extracted caries-free human third molars were selected for the study and stored in a solution of distilled water and thymol 0,2% at 4°C for up to 1 month after extractions, approved by the Ethics Committee in Research of the Piracicaba Dental School, State University of Campinas. The teeth were then scaled, cleaned, and stored in distilled water for 24 h. The teeth were randomly assigned to four experimental groups according to the composite resin (n=10).

Occlusal middle-depth dentin was exposed by sectioning the crowns parallel to the occlusal surface with a precision low-speed diamond saw (Isomet 1000, Buehler Ltd., Lake Buff, IL, USA) under water cooling. A dentin standard smear layer was created by polishing the occlusal surface with #600-grit SiC sandpaper for 60 s. The adhesives applied were according to their respective experimental groups (Table 2). Afterwards, the clinical crowns were restored with the composites using three increments of 2.0 mm each. Each increment was light-cured for 20 s (VIP Junior, BISCO, Schaumburg, IL, USA) for Heliomolar, Tetric N-Ceram, and Aelite LS and for 40 s for the Filtek Silorane composite, under irradiance of 600 mW/cm^2, which was constantly monitored with a radiometer. The teeth were stored at 37°C in distilled water for 24 h.

The restored specimens were then serially sectioned perpendicular to the adhesive-tooth interface at a 1.0 mm thickness using a slow-speed diamond saw. Approximately 4 toothpicks were tested immediately and 4 were stored for 1 year in distilled water, which was changed weekly. The specimens were tested individually by attaching them to a microtensile jig using cyanoacrylate glue (Super Bonder, Henkel/Loctite, Itapevi, SP, Brazil). The sticks were submitted to a tensile load using a universal testing machine (EZ Test, Shimadzu Corp., Kyoto, Japan) at a 1.0 mm/min cross-head speed. A digital caliper (Mitutoyo Corp., Kanagawa, Japan) was used to measure the bonding area in square millimeters.

The load in KgF and the bonding surface area of the specimen were

registered and microtensile bond strengths calculated in MPa. Statistical analysis was performed with statistical software (MINITAB 15, State College, PA, USA). Two-way analysis of variance (ANOVA) for the restorative systems and storage time factors was performed, followed by a Tukey's post hoc test ($p < 0.05$).

The fractured surfaces of the tested specimens were sputter-coated with gold (MED 010, Balzers, Balzer, Liechtenstein) and examined using a scanning electron microscope (VP 435, Leo, Cambridge, England). Failure patterns were classified as: Type I – adhesive failure; Type II – mixed failure; Type III – cohesive failure within dentin; and Type IV – cohesive failure within composite resin.

RESULTS

Polymerization stress measurements:

Table 3 presents the means of the polymerization stress for the composites and the ANOVA-detected statistical difference between them ($p = 0.003$). After five minutes of measuring, the restorative composites Tetric N-Ceram and Aelite LS showed lower polymerization stress means than those obtained for the Heliomolar composite. Filtek Silorane did not differ among all composite resins.

Internal adaptation analysis:

Table 4 presents the data in percentages of gaps of the restoration perimeter. Three-way ANOVA found statistical differences for the following factors: restorative systems ($p < 0.001$), storage time ($p = 0.003$), and restorative technique ($p < 0.001$). The values of gaps ranged from 4.6 to 66.1% (for composites Filtek Silorane and Aelite LS, respectively).

The gap formation was higher for the bulk-filling technique than for the incremental placement technique, independent of the restorative system tested. The gaps occurred mainly at gingival-axial line angle. The initial percentages of

gaps along the perimeter of the restorations were lower than those observed after 1 year of storage in distilled water.

The Filtek Silorane composite resin exhibited the lowest percentage of gaps when compared to other restorative systems (Figures 1 and 2), in terms of both evaluation times (24 h and 1 year) and composite placement techniques (incremental and bulk) ($p < 0.05$). The percentage of gap formation for the Aelite LS (Figures 3 and 4) composite did not differ for Heliomolar (Figures 5 and 6) and Tetric N-Ceram ($p > 0.05$) (Figures 7 and 8); however, Heliomolar showed higher gap formation than Tetric N-Ceram ($p < 0.05$).

Bond strength test:

Table 5 presents the mean bond strengths for the restorative systems after the two storage times. Two-way ANOVA revealed significant differences for the restorative systems factor ($p < 0.001$) and for the storage time factor ($p = 0.003$). Initially, the composite Filtek Silorane showed the lowest mean bond strength among the restorative systems; however, after storage for 1 year, this material showed the highest mean bond strength to dentin. At baseline, the Heliomolar/ExciTE, Aelite LS/One-Step Plus, and Tetric N-Ceram/ExciTE composites showed no significant difference in bond strength among them ($p > 0.05$). After storage for 1 year, the Heliomolar/ExciTE restorative system yielded lower bond strength than was observed for Aelite LS/One-Step Plus and Tetric N-Ceram/ExciTE materials ($p< 0.05$). The bond strength of the Filtek Silorane restorative system was unique in that it was not reduced after storage in water for 1 year ($p > 0.05$).

The total-etch adhesives produced a preponderance of mixed failures for both storage times (Figure 9). At baseline, most of the specimens bonded with silorane self-etching adhesive failed near or at the interface between the adhesive and dentin (Type I). After storage for 1 year, the restorative systems presented more cohesive failures in dentin (Type III).

DISCUSSION

Regarding polymerization stress, the Filtek Silorane restorative system did not differ from methacrylate-based composites, thus null hypothesis (1) was accepted. The silorane-based composite presented intermediate polymerization stress values for Heliomolar, Tetric N-Ceram, and Aelite LS, and this can be explained by the flexural modulus of the material, which is initially high in the pre-gel polymerization.[4,10,13] In this phase, the active species presents enough mobility to re-arrange and compensate for the volumetric shrinkage without generating significant amounts of internal and interfacial stresses.[14,15] Although Filtek Silroane showed a polymerization stress similar to that of methacrylate-based materials, Boaro et al. (2010)[13] found high polymerization stress values and low volumetric shrinkage (both post-gel and total) for this material.

For the Aelite LS composite resin, the results suggested that the addition of a high amount of filler is not an efficient approach for reducing the polymerization stress. The high content of filler particles in Aelite LS has somewhat controversial effects on shrinkage patterns. An increase of filler concentration by volume leads to reduced volumetric shrinkage as the resin volume is minimized. However, a high filler volume results in a stiff material with high elastic modulus that cannot efficiently absorb polymerization stresses.[17]

The results of the present investigation showed that no tested restorative systems (composite/adhesive) exhibited gap-free restorations. The continued polymerization shrinkage in association with elastic modulus generates stresses within the material, at the tooth/restoration interface, and within the tooth structure.[18,19] This stress state is likely to facilitate gap formation, which may reduce the longevity of the restoration.[17] Also, the integrity of the bonded interface depends on the interaction between shrinkage, elastic modulus, and adhesion to tooth structure.[20,21] Despite the fact that the manufacturer described the composite Aelite LS as being a low-shrinkage composite resin, the high amount of fillers, which did not result in low gap formation when compared to Tetric N-Bond, shows that it is a regular composite. These results can be

explained by their high flexural modulus, which interferes with the polymerization stress.[13]

Silorane-based restorations showed better internal adaptation than methacrylate-based restorations regardless of the restorative technique used and storage time, and thus null hypothesis (2) was rejected. The decreased gap formation may be associated with the low post-gel shrinkage of these composites, which has already been reported,[13,21,22] and the type of adhesive system used in combination with this composite, which contains a self-etch hydrophilic primer and a hydrophobic bonding resin.[23] Silorane System Adhesive is a two-step adhesive that has been categorized as a mild self-etch adhesive based upon its interaction with dentin up to a depth of a few hundred nanometers.[23] The primer and adhesive resin adhesive solutions were separately cured, resulting in typical two-fold bonding layers, which act as an elastic buffer.[24,25] that partially compensates for the shrinkage stress developed during polymerization of composites. The double adhesive layer and the highly hydrophobic nature of Silorane System Adhesive[23,26,27] were important not only for obtaining better internal adaptation and dentinal sealing but also to resist hydrolitic degradation during the water-storage for 1 year.

In the present study, filling the cavity using an incremental technique reduced the internal gap formation when compared to a bulk-filling restorative technique. Previous studies using conventional composites have already demonstrated that an incremental technique has a beneficial effect on the bond strength.[7,28,29] This positive effect should be attributed to reduce shrinkage stress by decreasing the C-factor of each layer. The internal adaptation of the rather stiff silorane composite improved with the incremental technique and the curing at the interface level might have been made more efficient by an increase in irradiance power (light is only dimmed by a thin layer of composite) and curing time (the total curing time was increased as well). A study by Van Ende et al. (2010)[30] examined the stress at the adhesive interface with different configuration factors, and their results indicated that cavity configuration affected the microtensile bond strength of the Silorane Adhesive System and considered that

an incremental layering technique is still required for placement of silorane composite restorations.

The high incidence of gaps formation was observed at the gingival-axial line angles, since there is an accumulation of bonding agents in this line angle, a difficult adaptation of composite in this location and tendency of high-stress in this areas.[31] After 1 year, all restorative systems tested showed significant increases in the gap formation and debonded margins. These gaps may result from water-induced degradation, which seemed to happen with methacrylate-based composite restorations. The composite water-induced degradation occurs by water absorption that causes monomer elution[32] or by degradation of the silane at the filler-monomeric network interface.[33] In contrast, silorane-based restorations also had the best marginal stability after 1 year of water storage. The combination of adhesive and composite, i.e., the good interaction with dentin and hydrophobicity for silorane, was responsible for such results.[10,11]

Total-etch adhesives exhibited the highest mean bond strength at the baseline, as previously reported.[34,35] This study found a significant reduction in bond strength for all total-etch adhesives after the aging treatment, evidenced by hydrolytic degradation over time. The weakening of the physical properties of the methacrylate-based resin-dentin-bonded interfaces occurs by chemical reactions of degradation of polymers and exposed collagen fibrils at the base of the hybrid layer.[36,37]

The dentin bond strength for the Filtek Silorane System Adhesive was the lowest at baseline, but after 1 year of storage in water, it was stable and the highest among the materials. Considering this result, null hypothesis (3) was rejected. The primer solution of Filtek Silorane Adhesive presented a pH 2.7 that provided a mild superficial demineralization of the tooth structure and the functional monomer seem to present chemical bonding to the hydroxyapatite crystals,[23] The hydrophobicity of the adhesive system may endow bonding layer hydrolytic-resistant characteristics with low water sorption,[23] leading to a mechanical stability of the adhesive interface.[36,38]

The failure patterns related for each group depended on the adhesive

system used or their interaction with the composite resin (Figure 9). Initially, the experimental groups that used total-etch adhesives showed mixed failures. For all materials, after 1 year of water storage, a slightly higher percentage of cohesive failure in the dentin and composite (Type III and IV) was reported, suggesting the degradation of the collagen matrix of dentin or the monomeric components of the composites that occurred in large numbers in the group using Heliomolar composite. Most Filtek Silorane specimens failed near the bonded interface between the adhesive layer and composite, suggesting that this interface is the weakest link. This finding is a reminder of the difference in nature between the silorane composite and the adhesive that connects it with the tooth structure.

CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn:

1. Compared to methacrylate-based composites, a silorane-based composite did not show significant polymerization stress reduction.
2. The silorane-based composite showed better internal adaptation than those observed for other restorative systems for both storage times.
3. The mean bond strength for the silorane-based composite remained stable after 1 year of storage in distilled water.

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TABLES

Table 1: Materials, manufacturer, composition, and batch number of the composites used in this study (information supplied by the MSDS of the manufacturer).

Material (Manufacturer)	Composition	Batch Number
Heliomolar® (Ivoclar Vivadent, Schaan, Liechtenstein)	BisGMA, UDMA, 1,10-decanediol dimethacrylate, camphorquinone, silicon dioxide, ytterbium trifluoride and prepolymerized filler (prepolymers) (46% vol.)	K35053
Tetric N-Ceram® (Ivoclar Vivadent, Schaan, Liechtenstein)	Dimethacrylates, additives, catalysts, stabilizers and pigments, barium glass, ytterbium trifluoride, mixed oxide and prepolymerized filler (prepolymers) (56% vol.)	L48183
Aelite™ LS (Bisco Inc., Schaumburg, IL, USA)	BisGMA, BisEMA, TEGDMA, camphorquinone, glass filler, amorphous sílica (74 vol%)	0900005990
Filtek™ Silorane (3M ESPE, St. Paul, MN, USA)	Bis-3,4-epoxycyclohexylethyl-phenyl-Methylsilane 3,4 Epoxycyclohexylcyclopolymethylsiloxane, camphorquinone, iodonium salt and electrondonor, silanized quartz, yttrium fluoride (55 vol%)	N205711

Abbreviations: bis-phenol A diglycidyl methacrylate (BisGMA), urethane dimethacrylate (UDMA), ethoxylated bisphenol A dimethacrylate (BisEMA) and triethylene glycol dimethacrylate (TEGDMA).

Table 2: Materials, manufacturer, composition, and batch number of the adhesives used in this study (information supplied by the MSDS of the manufacturer).

Material (Manufacturer)	Composition	Batch Number	Directions for uses
One-Step® Plus (Bisco, Inc., Schaumburg, IL USA)	Biphenyl dimethacrylate, hydroxyethyl methacrylate, acetone, dental glass	0800005538	Apply phosphoric acid gel 37% to the prepared dentin for 15 s. Remove the gel with a vigorous water spray for 15 s. Remove the excess moisture with foam pellet (wet bonding). Shake bottle once. Apply 2 generous coats to the preparation. Agitate lightly for 10–15 s. Dry gently for 5 s. Cure for 10 s
ExciTE® (IvoclarVivadent, Schaan, Liechtenstein)	Bisphenol A glycol dimethacrylate, ethanol, 2-hydroxyethyl methacrylate, phosphonic acid acrylate, urethane dimethacrylate	L31463	Apply phosphoric acid gel 37% to the prepared dentin for 15 s. Remove the gel with a vigorous water spray for 15 s. Remove the excess moisture with foam pellet (wet bonding). Apply the adhesive on the prepared surfaces for at least 10 s. Dry gently for 5 s. Cure for 10 s
Filtek Silorane System Adhesive (3M ESPE, St. Paul, MN, USA)	*Self-etching primer:* phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, silane-treated silica filler, initiators, stabilizers *Bond:* hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers	*Primer:* N209848 *Adhesive:* N204592	Shake bottle briefly. Apply visibly thick layer. Gentle air dispersion until movement stops. Cure for 10 s Gently dry surface. Apply and leave undisturbed for 10 s. Then, dry for 5 s with maximum air pressure. Cure for 10 s

Table 3. Mean polymerization stresses (standard deviation)of the composite resins used in this study (in MPa).

Resin composites	Polymerization stress (MPa) (SD)
Heliomolar®	2.3 (0.4) A
Aelite™ LS	2.3 (0.7) A
Filtek™Silorane	2.7 (0.5) AB
Tetric N-Ceram®	3.2 (0.6) B

Means followed by different letters are significantly different.

Table 4. Mean percentages of gaps for restorative systems following the restorative placement techniques and storage times.

	Incremental		***Bulk***	
	24 hours	***1 year***	***24 hours***	***1 year***
Tetric N-Ceram®	21.9 Ba*	37.2 Bb*	30.2 Ba	50.5 Bb
Heliomolar®	37.4 Ca*	51.4 Cb*	48.7 Ca	61.0 Cb
Aelite™ LS	27.8 BCa*	36.1 BCb*	51.4 BCa	66.1 BCb
Filtek™Silorane	4.9 Aa*	20.8 Ab*	6.6 Aa	22.9 Ab

Means followed by different letters represent significant differences (3-way ANOVA and Tukey's test, $p < 0.05$). Capital letters compare composites within the same technique and time; lower case compare the storage times within the same placement technique and composite; symbols (*) represent differences between composite placement technique.

Table 5. Mean bond strengths (standard deviation) after 24 hours and 1 year of storage (in MPa).

	Bond Strength	
Composite/Adhesive	*24 hours*	*1 year*
Heliomolar/Excite	51.6 (6.8) Aa	23.1 (4.4) Cb
Aelite LS/One-Step Plus	48.4 (3.9) Aa	27.8 (3.2) Bb
Tetric N-Ceram/Excite	47.0 (2.9) Aa	28.5 (2.7) Bb
Filtek Silorane/Silorane Adhesive	37.3 (4.3) Ba	36.1 (2.1) Aa

Means followed by different letters (uppercase - column; lower case - row) are significantly different.

FIGURES

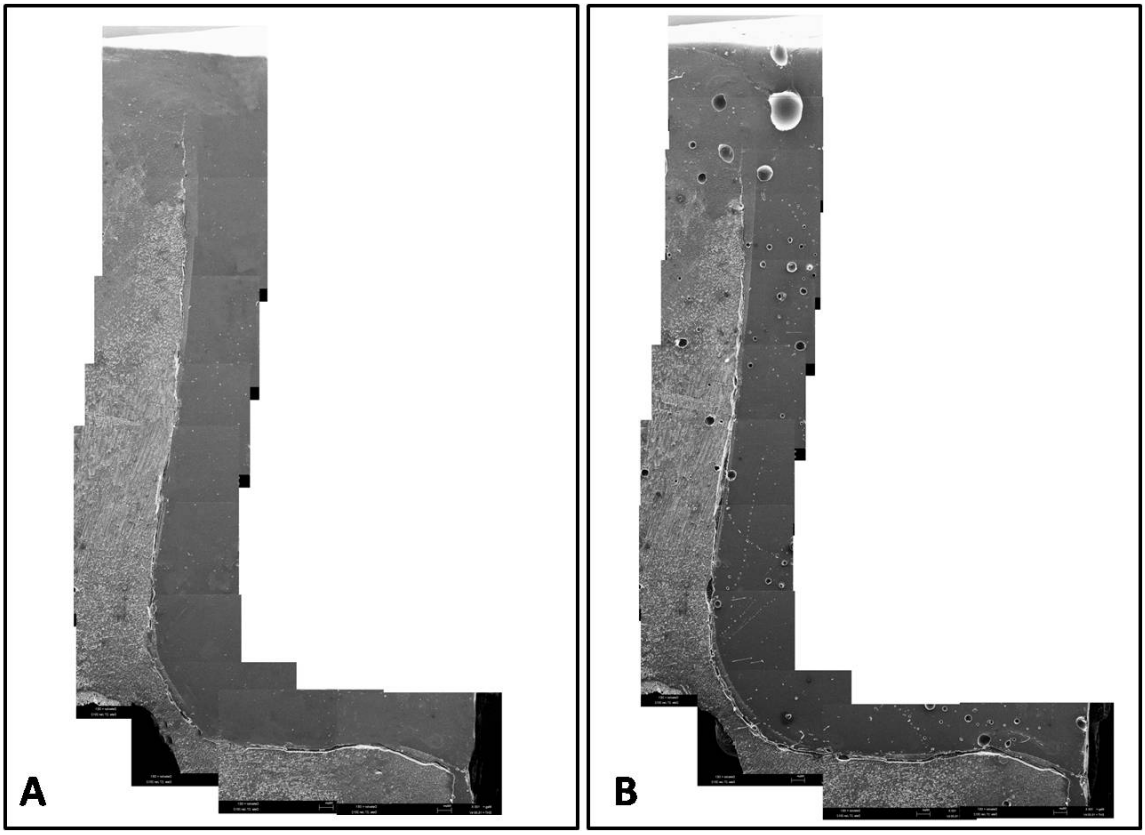

Figure 1. Photomicrograph of the internal interface of Class II cavity restored with Tetric N-Ceram using a bulk-filling technique: (A) 24 h of storage and (B) 1 year of storage.

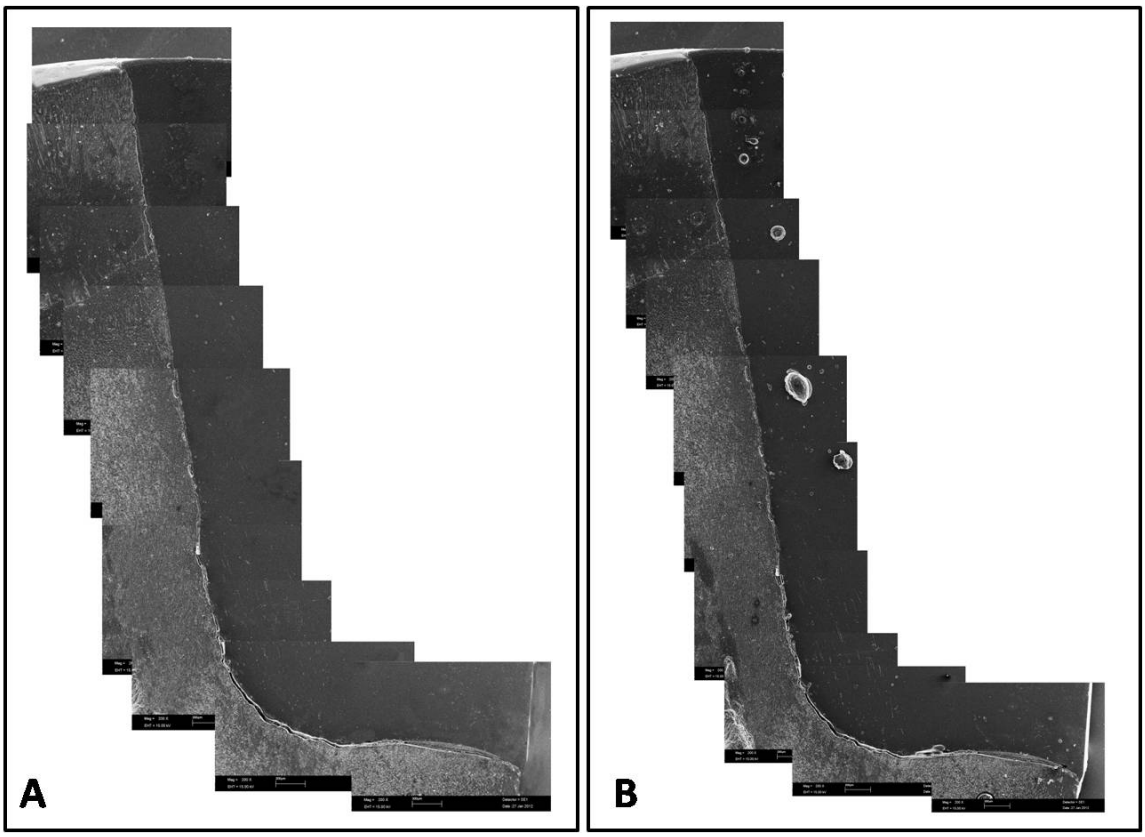

Figure 2. Photomicrograph of the internal interface of Class II cavity restored with Tetric N-Ceram using oblique incremental filling technique: (A) 24 h of storage and (B) 1 year of storage.

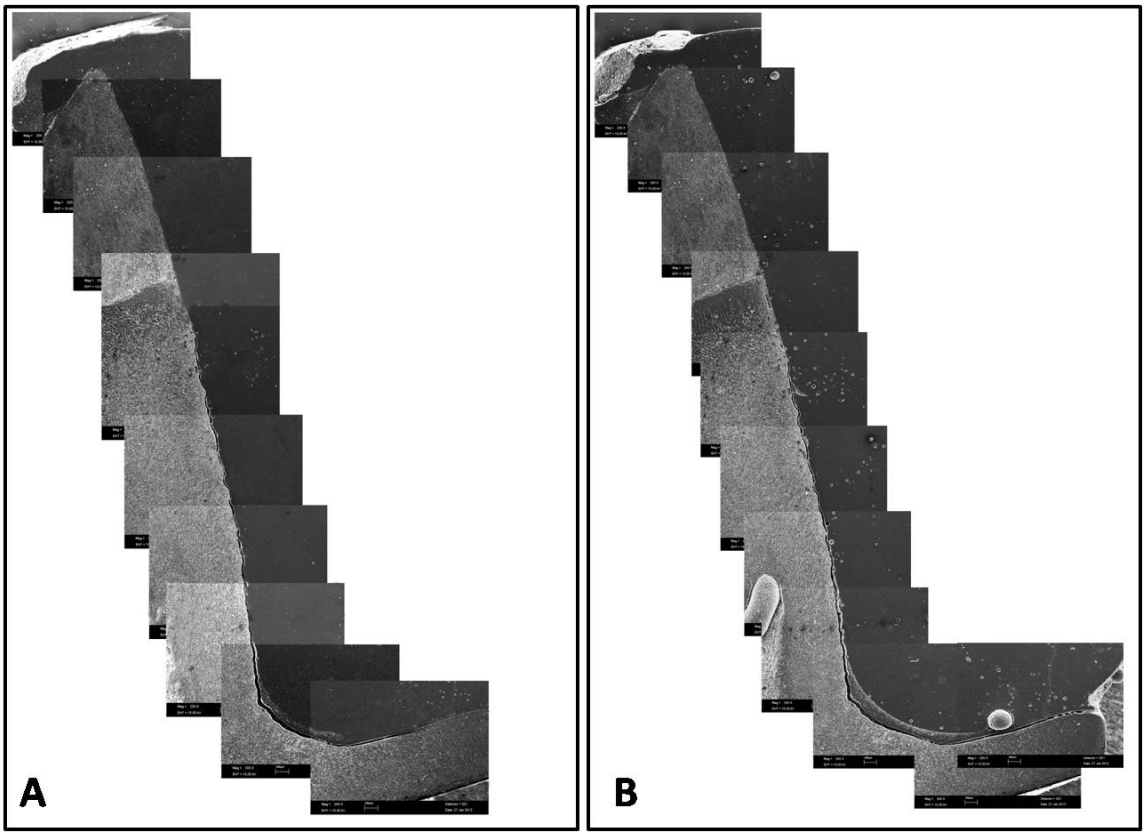

Figure 3. Photomicrograph of the internal interface of Class II cavity restored with Heliomolar using a bulk-filling technique: (A) 24 h of storage and (B) one year of storage.

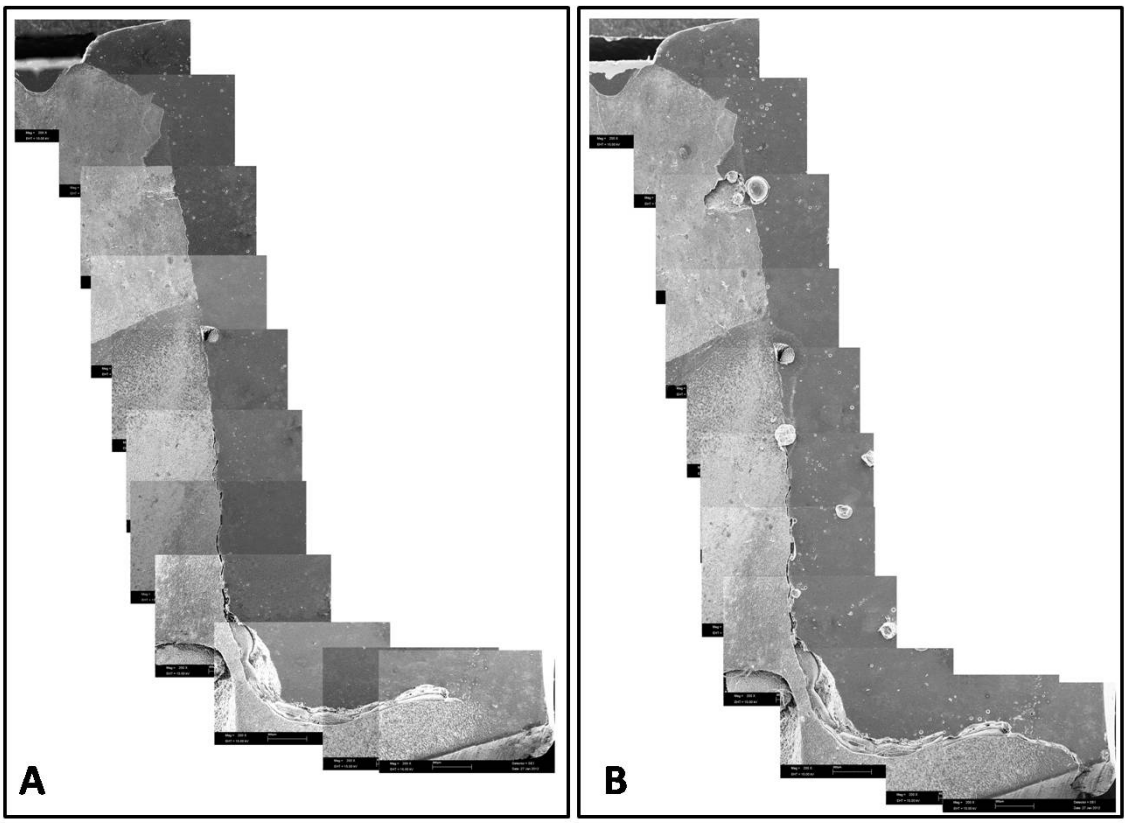

Figure 4. Photomicrograph of the internal interface of Class II cavity restored with Heliomolar using oblique incremental filling technique: (A) 24 h of storage and (B) 1 year of storage.

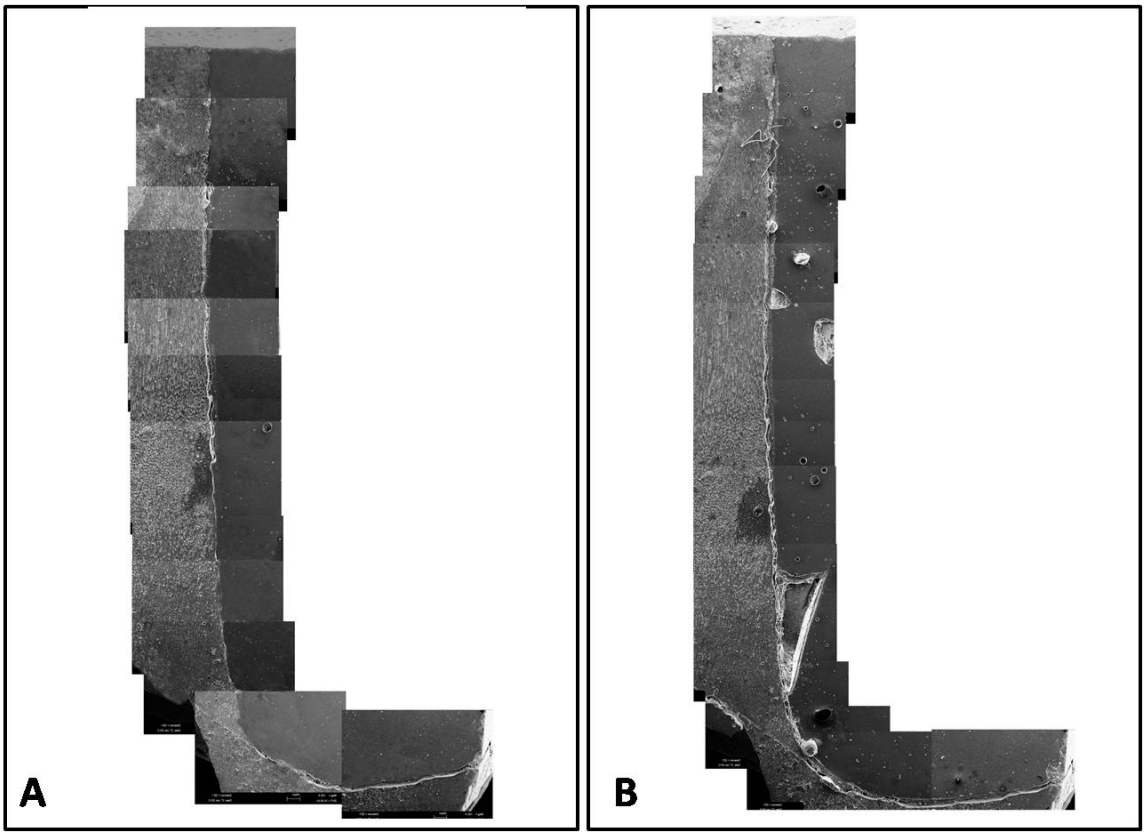

Figure 5. Photomicrograph of the internal interface of Class II cavity restored with Aelite LS using a bulk-filling technique: (A) 24 h of storage and (B) 1 year of storage.

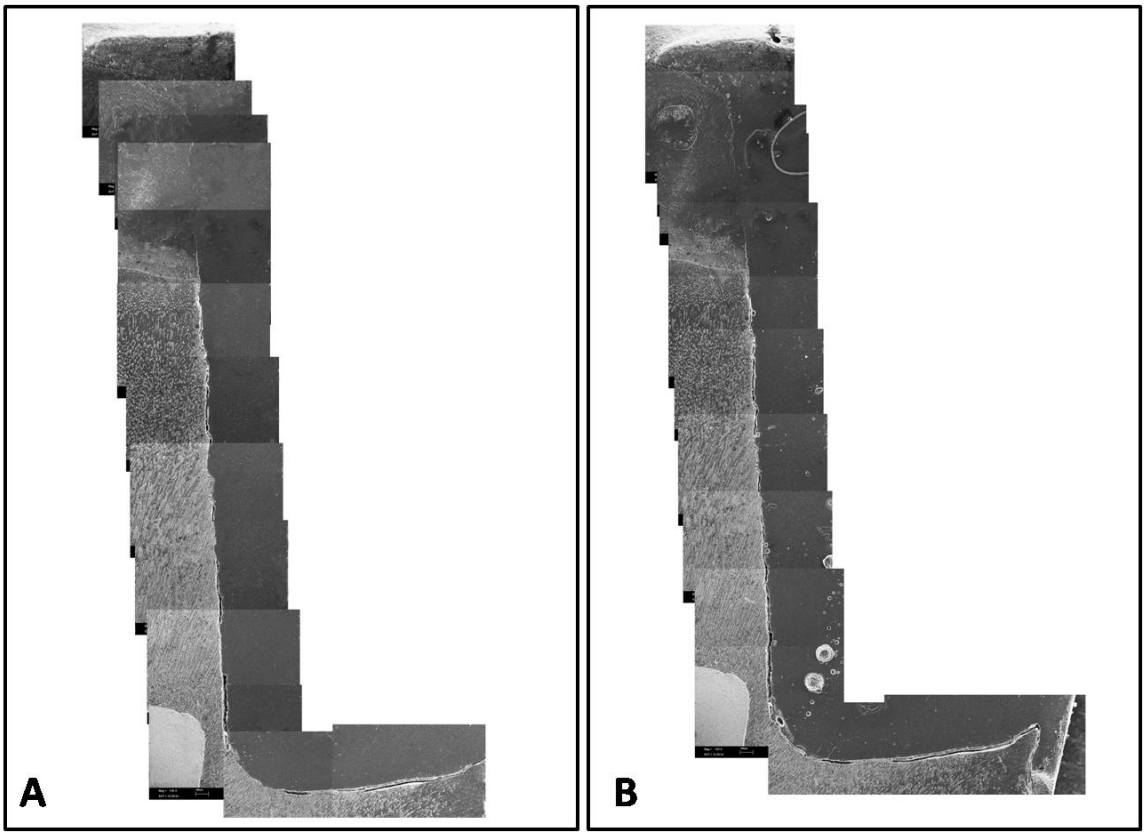

Figure 6. Photomicrograph of the internal interface of Class II cavity restored with Aelite LS using oblique incremental filling technique: (A) 24 h of storage and (B) 1 year of storage.

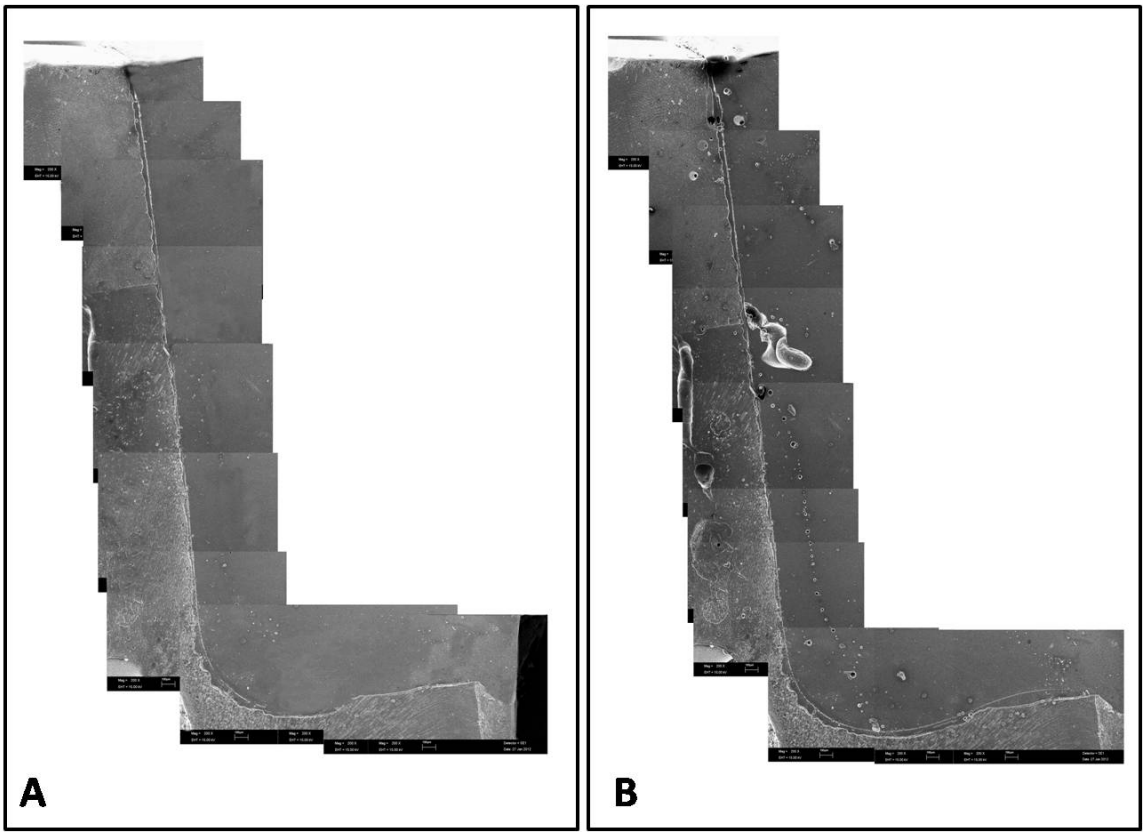

Figure 7. Photomicrograph of the internal interface of Class II cavity restored with Filtek Silorane using a bulk-filling technique: (A) 24 h of storage and (B) one year of storage.

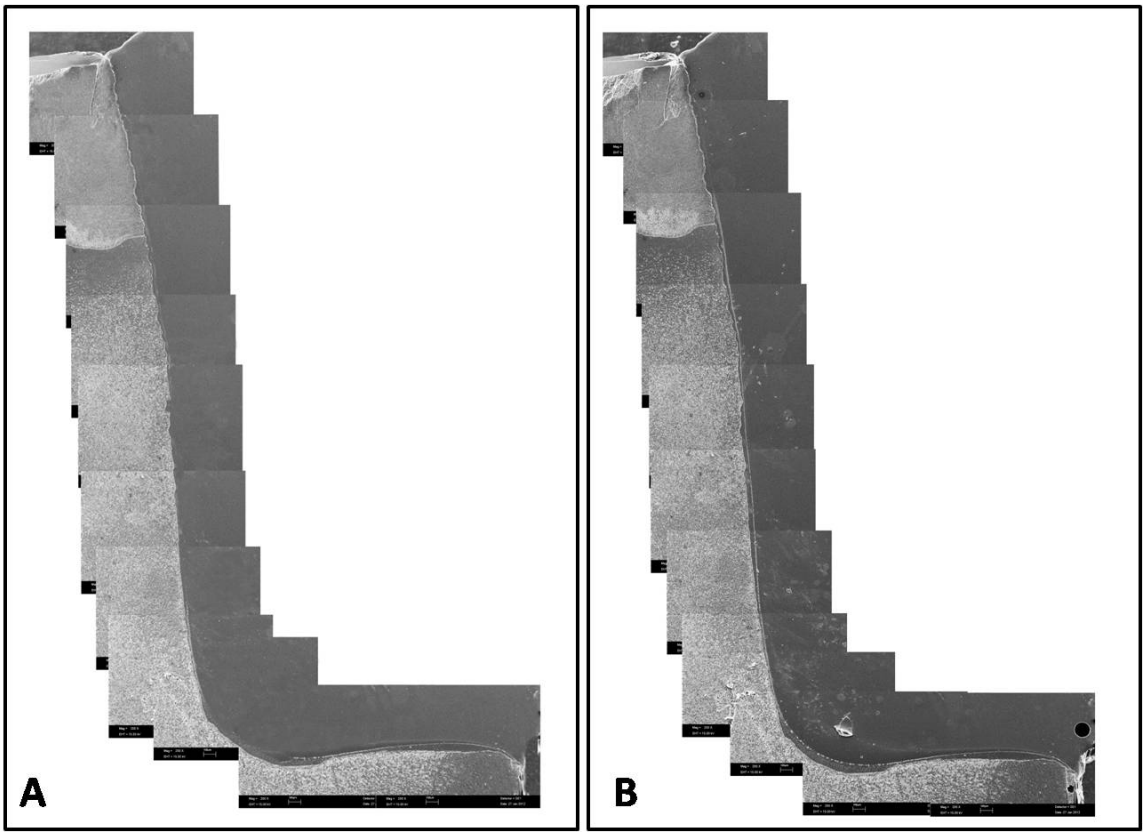

Figure 8. Photomicrograph of the internal interface of Class II cavity restored with Filtek Silorane using oblique incremental filling technique: (A) 24 h of storage and (B) 1 year of storage.

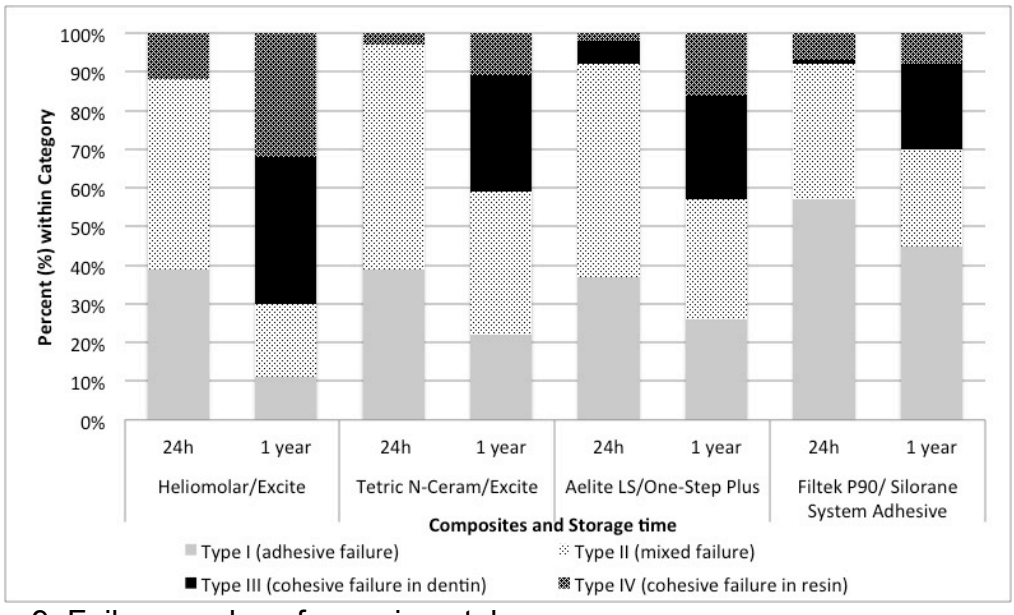

Figure 9. Failure modes of experimental groups.

CONSIDERAÇÕES GERAIS

As resinas compostas de baixa contração foram disponibilizadas no mercado odontológico com o intuito de reduzir a contração volumétrica durante a polimerização e seus efeitos deletérios. Através da modificação na formulação dos materiais restauradores tanto na parte monomérica (Filtek Silorane) quanto na quantidade de partículas de carga para a Aelite LS, os fabricantes alegam que essas alterações são capazes de promover redução na tensão de contração do material. Informações detalhadas quanto às propriedades mecânicas, o mecanismo e a durabilidade da união desses materiais representam um tópico relevante e atual da Odontologia Restaurada, principalmente quando comparados às resinas compostas de formulações convencionais. Assim, este estudo avaliou 4 resinas compostas (2 de baixa contração e 2 convencionais) quanto à composição inorgânica e às características morfológicas das partículas de carga, utilizando MEV/EDS; tensão de polimerização, analisou a rugosidade superficial dos materiais após acabamento e polimento, a sorção e solubilidade, a formação de fendas internamente em cavidades Classe II restauradas com técnica incremental e bloco único, e resistência de união à dentina dos sistemas restauradores. Adicionalmente, foi avaliado o efeito do armazenamento por 1 ano em água destilada para os testes acima realizados, exceto para a análise das partículas de carga e da tensão de polimerização.

Na avaliação das partículas de carga buscou-se identificar os principais componentes inorgânicos e caracterizar essas partículas presentes nas resinas compostas. A resina compostas Aelite LS, que apresenta cerca de 76% de partículas de carga em volume, contém partículas esféricas (de 0,5 a 3 μm), que pode ser sílica e outras irregulares, que sugerem ser partículas de vidro (5 a 10 μm). A forma esférica dessas partículas permite melhorar o empacotamento das partículas na matriz (Bayne *et al.*,1994) e, portanto, permite um aumento do volume de partículas no material (Hosoda, *et al.*,1990; Khan, 1992).

O compósito Filtek Silorane, um compósito de baixa contração que possui silorano como componente monomérico, mostrou partículas de quartzo e fluoreto de ítrio com formato irregulares de tamanho máximo de 1 μm. O quartzo apresenta alta dureza e menor dissolução que a sílica coloidal (Sherwoodv, 2010). Devido à falta de opacidade do quartzo, a adição do fluoreto de ítrio é necessária (Abers, 2002).

Na avaliação da rugosidade superficial, utilizamos os sistemas de polimento do mesmo fabricante da resina composta testada, e as diferentes formas e materiais de polimento podem ter influenciado nos resultados apresentados. Sistemas de polimentos como as pontas abrasivas Astropol (Ivoclar Vivadent) e Finishing Discs (Bisco Inc.) promovem abrasividade na superfície da resina, enquanto os discos Sof-Lex (3M ESPE) promovem um corte da superfície, em granulações maiores, e o polimento nas menores granulações (Reis *et al.,* 2002). Isso pode ter levado aos menores valores obtidos de rugosidade superficial para a resina composta Filtek Silorane, mesmo possuindo em sua composição partículas irregulares de quartzo. O pequeno tamanho médio dessas partículas (máximo de 1 μm) e a baixa solubilidade do silorano e baixa dissolução do quartzo também contribuíram para os resultados, que se mantiveram superiores mesmo após 1 ano de armazenamento. As demais resinas compostas apresentaram valores superiores inicialmente e após 1 ano de armazenamento, devido possivelmente ao sistemas de polimento utilizados, da matriz orgânica de metacrilatos, tamanho e formato das partículas de carga, que são muito irregulares em formato e tamanho para o compósito Heliomolar e em grande quantidade em Aelite LS. Embora os bons resultados obtidos, o uso do sistema Sof-Lex juntamente com a Filtek Silorane não é viável clinicamente, pois o compósito é destinado para restaurações de dentes posteriores.

A sorção e solubilidade de resinas compostas dependem diretamente da sua composição (Toledano *et al.,* 2003). Os baixos valores de sorção para Aelite LS, quando comparados com as demais resinas compostas deve-se à alta quantidade de partículas de carga (74% vol.) (Pearson & Longman, 1989). Os

valores de sorção e solubilidade da Filtek Silorane foram inferiores tanto inicialmente quanto após 1 ano, devido à característica hidrofóbica do silorano (Palin *et al.,* 2005; Janda *et al.,*2007).

Embora as resinas compostas Aelite LS e Filtek Silorane serem de baixa contração, os valores obtidos de tensão de polimerização não foram inferiores quando comparados aos da resina Heliomolar e Tetric N-Ceram, os quais apresentam valores semelhante às resinas com formulações convencionais. Filtek Silorane apresenta um alto módulo flexural na fase pré-gel da polimerização, aonde as moléculas não conseguem apresentar uma mobilidade para compensar o estresse de polimerização (Weinmann *et al.,* 2005; Lee *et al.,* 2007;Boaro *et al.,*2010). Aelite LS, devido sua alta concentração de partículas de carga, apresenta um alto módulo de elasticidade, que impede uma eficiente absorção da tensão de polimerização (Lopes *et al.*,2004).

Quanto ao estudo da formação interna de fendas, nenhum sistema restaurador apresentou ausência total de fendas. A integridade da interface de união está diretamente associada à interação entre a tensão de contração, módulo de elasticidade e a adesão à estrutura dental (Papadogiannis *et al*,2009; Takahashi*et al,*2010). Como discutido anteriormente, o alto módulo de elasticidade da resina Aelite LS interfere no estresse de polimerização, resultando em formação de fendas semelhante à Tetric N-Ceram. As restaurações confeccionadas com o sistema contendo silorano mostraram melhor adaptação interna, mesmo após o período de armazenamento. A resina composta Filtek Silorane necessita de um sistema adesivo específico, autocondicionante, onde o *primer* de propriedade hidrofílica é fotopolimerizado, seguida da aplicação de um *Bond* hidrofóbico. Essas soluções polimerizadas separadamente, resultam em duas camadas adesivas, de relativa espessura, que, mesmo com tensão de polimerização semelhante aos demais compósitos, serve como um “colchão” elástico, absorvendo em parte as tensões geradas na interface. A natureza hidrofóbica tanto do sistema adesivo quanto da resina composta à base de silorano contribuíram para que as porcentagem de fendas mantenham-se inferiores mesmo após o período de 1 ano.

Em relação à técnica restauradora utilizada em restaurações de cavidades Classe II, a incremental é a que promoveu os menores valores formação de fendas, independente do sistema restaurador utilizado. Isso confirma achados anteriores (Reis *et al.*,2003; He *et al.*,2007; Nayif *et al.*,2008), que mostraram ser a técnica com melhores benefícios quando comparado à técnica de bloco único. Mesmo alegando ser possível confeccionar uma restauração em bloco único, a resina composta Filtek Silorane apresentou valores inferiores de fendas internas quando utilizou-se a técnica incremental.

Os sistemas adesivos com condicionamento total, como Excite (Ivoclar Vivadent) e One-Step Plus (Bisco Inc.) apresentaram os maiores valores iniciais (24 h) de resistência de união, que reduziram após o período de armazenamento, sugerindo degradação dos componentes da união dentina-resina(Hashimoto *et al.*, 2003; Breschi *et al.,* 2008). Embora inicialmente a resistência de união do Filtek Silorane System Adhesive (3M ESPE) tenha sido menor, essa união manteve-se estável após o período de armazenamento. O pH de 2,7 do primer autocondicionante é considerado fraco, no entanto especula-se sobre uma adesão química com os cristais de hidroxiapatita (Mine *et al*., 2010), e a características hidrofóbica e com baixa sorção e solubilidade do silorano, contribuiu para manter estável a união do adesivo com a dentina (Mine *et al*., 2010).

Diante dos resultados obtidos no presente estudo, os compósitos ditos de “baixa contração” mostraram-se, na maioria dos testes realizados, semelhantes às resinas compostas de formulações convencionais, no entanto os benefícios apresentados pela Filtek Silorane, como uma baixa sorção e solubilidade, baixa presença de fendas na interface interna das restaurações, e um sistema adesivo com boa estabilidade ao longo do tempo,sugere que este material é uma boa alternativa aos demais sistemas restauradores presentes no mercado Odontológico para a restauração de dentes posteriores.

CONCLUSÃO

A partir dos resultados obtidos nos três capítulos que compõem este estudo, pode-se concluir que:

- A caracterização de partículas de carga em microscópio eletrônico de varredura apresentou variações morfológicas entre as resinas compostas investigadas. A microanálise EDS também detectou diferenças na composição inorgânica, e o elemento silício estava sempre presente. Em geral, os componentes inorgânicos estavam de acordo com as informações fornecidas pelos fabricantes (MSDS);
- Filtek Silorane, material composto por silorano, apresentou menor rugosidade superficial, menor sorção e solubilidade que as resinas compostas à base de metacrilatos avaliadas neste estudo;
- O armazenamento em água durante um ano aumentou sorção para todas as resinas compostas testadas;
- Comparado às resinas compostas de formulação convencional, as resinas Filtek Silorane e Aelite LS (classificadas como de baixa contração de polimerização) não apresentaram redução significativa de estresse de polimerização;
- compósito baseado em silorano Filtek Silorane apresentou menor formação de fendas internas entre as resinas compostas estudadas em ambos os períodos de armazenamento em água destilada(24 horas e 1 ano);
- Os valores iniciais de resistência de união dos sistemas adesivos com condicionamento total foram inicialmente superiores, porém o sistema restaurador Filtek Silorane System Adhesive/ Filtek Silorane foi o único que permaneceu estável após um ano de armazenagem em água destilada.

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* De acordo com a norma da UNICAMP/FOP, baseada na norma do International Committee of Medical Journal Editors – Grupo de Vancouver. Abreviatura dos periódicos em conformidade com Medline.

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ANEXO

ANEXO - Certificado do Comitê de Ética em Pesquisa, FOP-UNICAMP;

COMITÊ DE ÉTICA EM PESQUISA
FACULDADE DE ODONTOLOGIA DE PIRACICABA
UNIVERSIDADE ESTADUAL DE CAMPINAS

CERTIFICADO

O Comitê de Ética em Pesquisa da FOP-UNICAMP certifica que o projeto de pesquisa **"Avaliação da resistência de união, contração de polimerização, adaptação em cavidade classe II e de outras propriedades de sistemas restauradores"**, protocolo nº 090/2009, dos pesquisadores Marcelo Giannini e Marina Di Francescantonio, satisfaz as exigências do Conselho Nacional de Saúde - Ministério da Saúde para as pesquisas em seres humanos e foi aprovado por este comitê em 08/07/2009.

The Ethics Committee in Research of the School of Dentistry of Piracicaba - State University of Campinas, certify that the project **"Evaluation of the bonding strength, polymerization contraction, adaptation in class II restorations and other properties of restorative systems"**, register number 090/2009, of Marcelo Giannini and Marina Di Francescantonio, comply with the recommendations of the National Health Council - Ministry of Health of Brazil for research in human subjects and therefore was approved by this committee at .

Prof. Dr. Pablo Agustin Vargas
Secretário
CEP/FOP/UNICAMP

Prof. Dr. Jacks Jorge Junior
Coordenador
CEP/FOP/UNICAMP

ANEXO 1- Confirmação de submissão de artigo referente ao Capitulo 1;

Submission Confirmation

Thank you for submitting your manuscript to *Journal of Microscopy*.

Manuscript ID:	JMI-2012-0197
Title:	Investigation of filler particle composition and morphology of low-shrinkage and traditional composite resins by SEM and EDX
Authors:	Di Francescantonio, Marina Pacheco, Rafael Aguiar, Thaiane Martins, Adriano Boaro, Letícia Braga, Roberto Giannini, Marcelo
Date Submitted:	28-Dec-2012

ANEXO 2- Confirmação de submissão de artigo referente ao Capitulo 2;

OPERATIVE DENTISTRY

ONLINE MANUSCRIPT SUBMISSION AND PEER REVIEW

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Detailed Status Information

Manuscript #	12-526-L
Current Revision #	0
Submission Date	2012-12-29
Current Stage	Initial QC Started
Title	Characterization of Water Sorption, Solubility, and Roughness of Silorane- and Methacrylate-Based Composite Resins
Running Title	Water sorption, solubility, and roughness of composite resins
Manuscript Type	Laboratory Research
Corresponding Author	Marcelo Giannini (Piracicaba Dental School, University of Campinas)
Contributing Authors	Marina Di Francescantonio , Rafael Pacheco , Letícia Boaro , Roberto Braga
Financial Disclosure	I have no relevant financial interests in this manuscript.
Abstract	Objective: The objective of this study was to evaluate the surface roughness (SR), water sorption (WS), and solubility (SO) of four composite resins after finishing/polishing and after one year of water storage. Material and Methods: Two low-shrinkage composites (Filtek Silorane, 3M ESPE and Aelite LS, Bisco Inc.) and two composites of conventional formulations (Heliomolar and Tetric N-Ceram, Ivoclar Vivadent) were tested. Their respective finishing and polishing systems (Sof-Lex Discs, 3M ESPE; Finishing Discs Kit, Bisco Inc., and Astropol F, P, HP, Ivoclar Vivadent) were used following the manufacturer's instructions. Ten disc-shaped specimens of each composite resin were made for each evaluation. Polished surfaces were analyzed using a profilometer after 24 h and one year. For the WS and SO, the discs were stored in desiccators until constant mass was achieved. Specimens were then stored in water for 7 days or one year, at which time the mass of each specimen was measured. The specimens were dried again and dried specimen mass determined. The WS and SO were calculated from these measurements. Data analyzed by two-way ANOVA and Tukey's post-hoc test (α = 0.05). Results: Filtek Silorane showed the lowest SR, WS, and SO means. Water storage for one year increased the WS means for all composite resins tested. Conclusions: The silorane-based composite resin results were better than those obtained for methacrylate-based resins. One-year water-storage did not change the SR and SO properties in any of the composite resins.
Key Words	composites resins, water sorption, solubility, roughness, silorane
Topic	FINISHING PROCEDURES FOR RESTORATIVE MATERIALS, OTHER/NOT LISTED
Clinical Relevance	This study showed that silorane-based composites demonstrated good performance in all parameters studied (water sorption, solubility, and roughness), suggesting its use as good alternative restorative material.
Copyright Release Date	Not Received

Stage	Start Date
Initial QC Started	2012-12-29
Author Approved Converted Files	2012-12-29
Preliminary Manuscript Data Submitted	2012-12-29

ANEXO 3- Confirmação de submissão de artigo referente ao Capitulo 3.

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Marina Di Francescantonio <marinadifr@gmail.com>

Submission Confirmation for Journal of Dentistry

1 mensagem

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Dear Dr. Di Francescantonio,

Your submission entitled "Internal adaptation analysis, bond strength, and polymerization stress of silorane- and methacrylate-based composites" has been received by the Journal of Dentistry.

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